# *Dynamic surface chemistry effects on lithium-coated graphite surfaces from deuterium irradiation*

J.P. Allain[1,2,3], C.N. Taylor[1], B. Heim[1], L. Kollar[1]

PPPL Collaborators: C.H. Skinner, H.W. Kugel, R. Kaita, A.L. Roquemore

*[1]Purdue University, West Lafayette, IN*

*[1]School of Nuclear Engineering*

*[2]School of Materials Engineering*

*[3]Birck Nanotechnology Center, Discovery Park, Purdue University*

Physics Meeting, Monday 3 August, 2009, Princeton Plasma Physics Laboratory

RSSEL

PURDUE UNIVERSITY

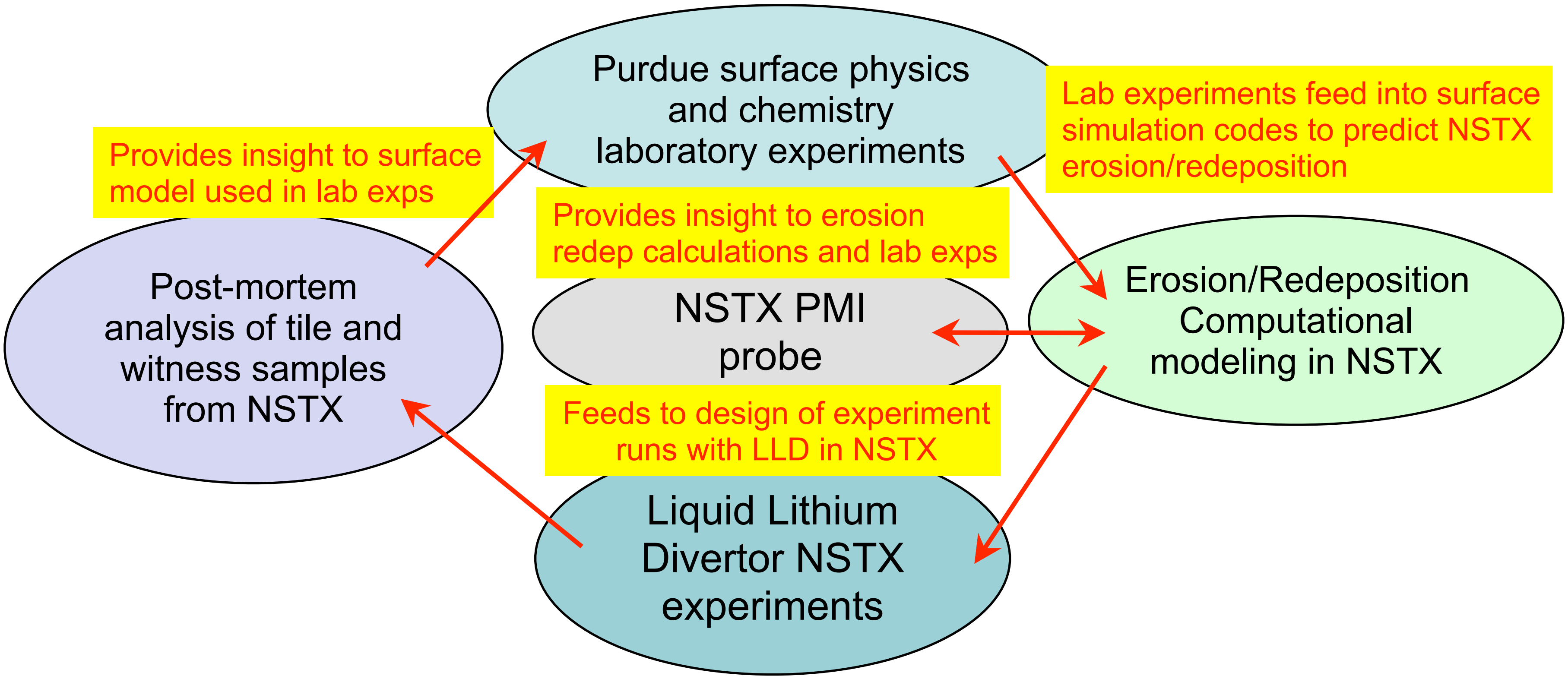
PURDUE UNIVERSITY Discovery Park

PURDUE ENGINEERING

# *Outline*

- Summary of Li-based PMI work at Purdue
- Lithium-based surfaces and D retention
- Post-mortem NSTX tile analysis
- Study of D-irradiated lithiated graphite
- PMI probe results
- Current issues of lithiated graphite work
- Implications for LLD operation
- Summary and Future Work

# Summary of Li-based PMI work at Purdue

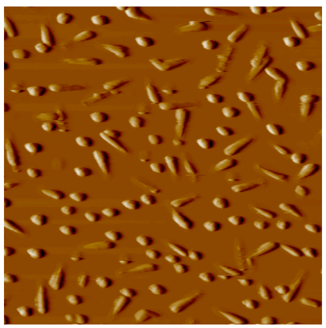


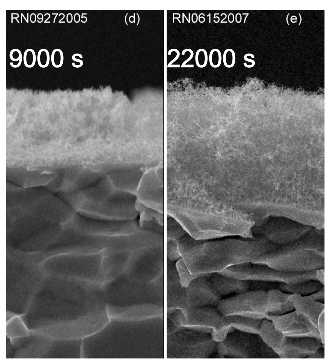

- At Purdue we're investigating the fundamental role lithium coatings on ATJ graphite play on deuterium pumping and recycling of hydrogen
- We systematically study lithiated graphite surface chemistry and ion-induced desorption to elucidate plasma-material interface interactions in NSTX
- Lab experiments also look at the effect of a lithiated graphite environment on the performance of NSTX plasma with the liquid lithium divertor (LLD)

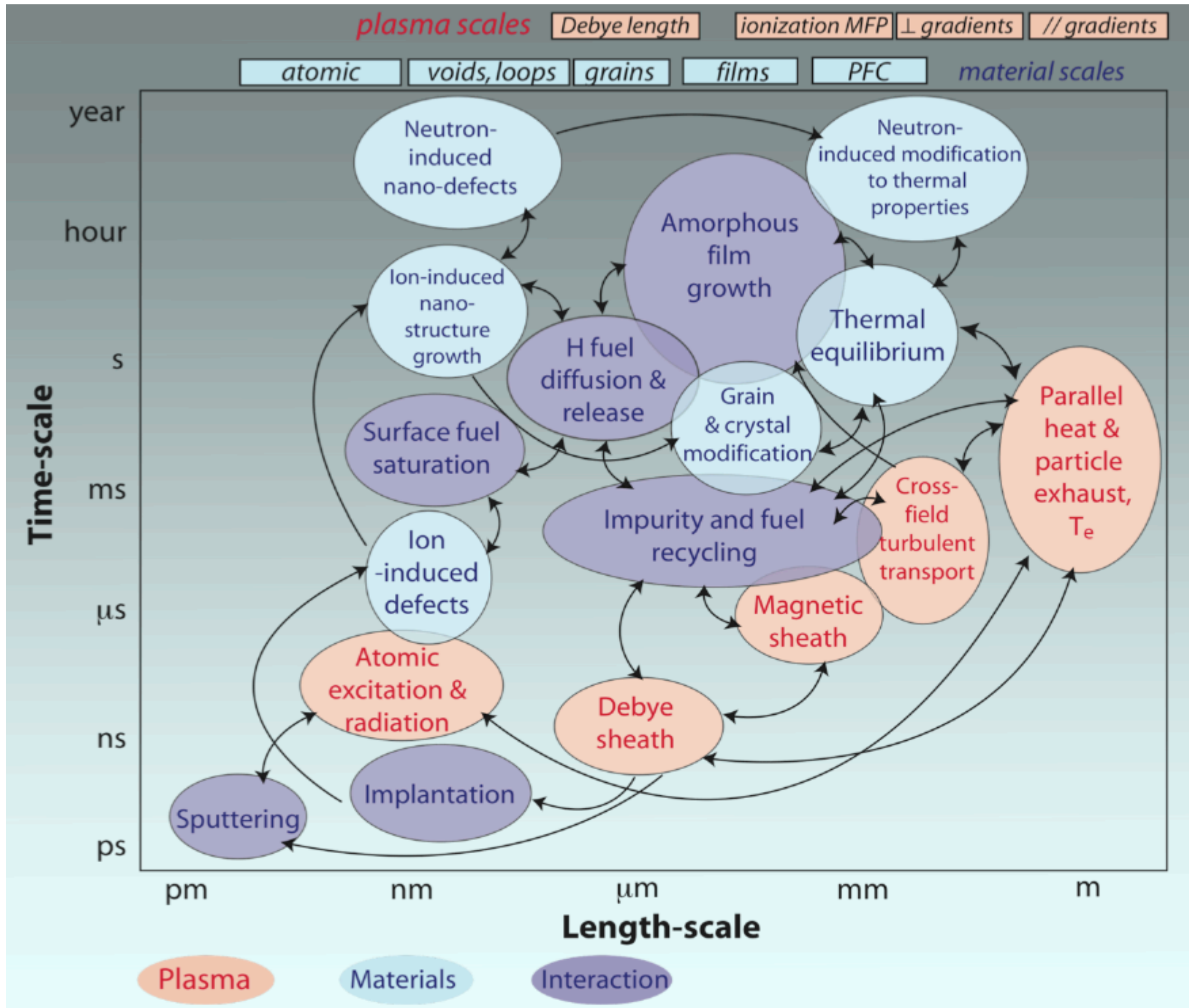
# *Lithiated graphite work at Purdue*

- Post-mortem analysis of 2008 NSTX campaign tiles
  - Along inner divertor floor and bottom of center stack tiles
  - Tiles near LiTER port
  - Also examined Si witness samples (retrieved from various locations in NSTX)
- Controlled *in-situ* lithiated graphite studies
  - Correlation of D irradiations with graphite tiles
  - Mechanisms for D retention as function of D flux and Li dose
  - Mechanisms for surface passivation on Li-C
  - Control experiments with: Si, lithium foil, SS, Mo, W, etc…
- NSTX PMI probe design and analysis
  - Probe samples: Si, ATJ graphite, Pd
  - TDS and XPS analysis

# *Radiation-driven vs naturally-driven systems: instabilities and self-organization at the plasma-surface interface*





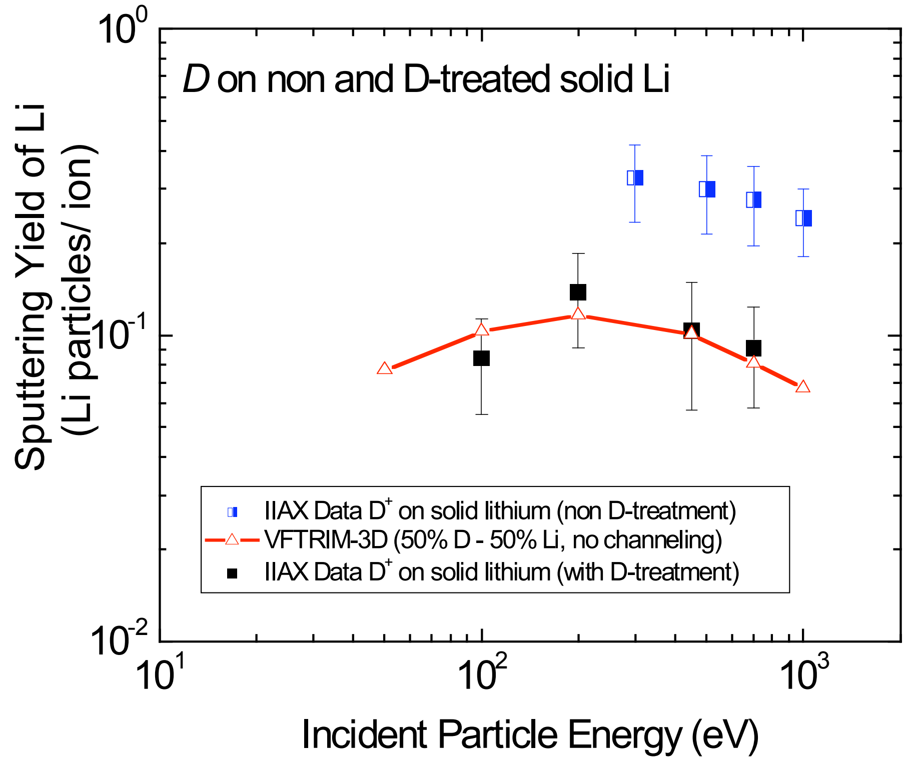

*Courtesy of: B. Wirth*

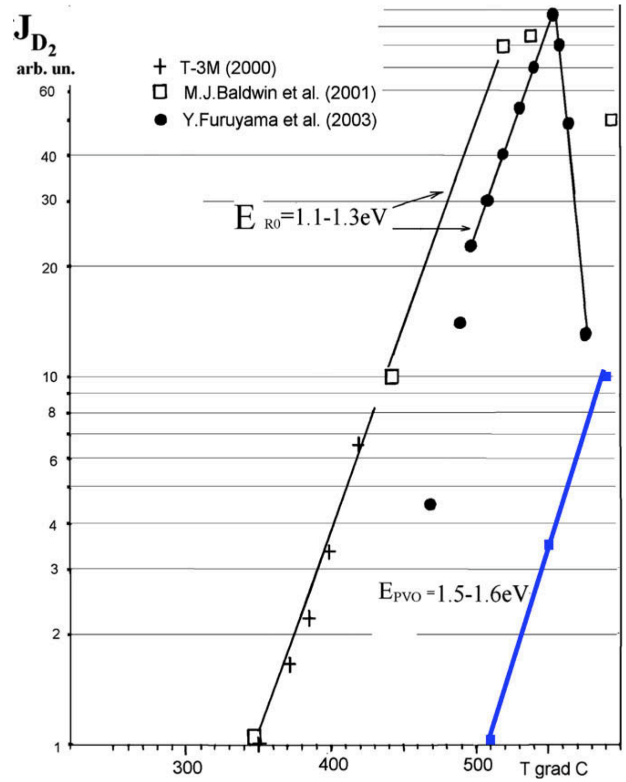
# *Lithium as a pump for hydrogen: the role of spatial scales*

- Critical to the recycling of D when using lithium as a PFS (plasma-facing surface) is the top layer of atoms
- Sputtered particles emanate from the first 2-3 ML of a metal surface (although the damage zone can be 10-100's nm below)
- Recombination of implanted D occurs at the first few layers at the surface-vacuum interface
- Diffusion and other mechanisms from surface-to-bulk and vice versa are obviously important, however we focus on the net condensed matter state *at* the surface
- Understanding the lithium surface properties (sputtering, D retention, ion yield, etc…) requires probing at these spatial scales

*J.N. Brooks and J.P. Allain, J. Nucl. Materials, 337-339 (2005) 1053*
*J.P. Allain, J.N. Brooks, Guojing Ho, J. Nucl. Mat. in preparation 2008*

# *Liquid lithium sputtering and D retention*



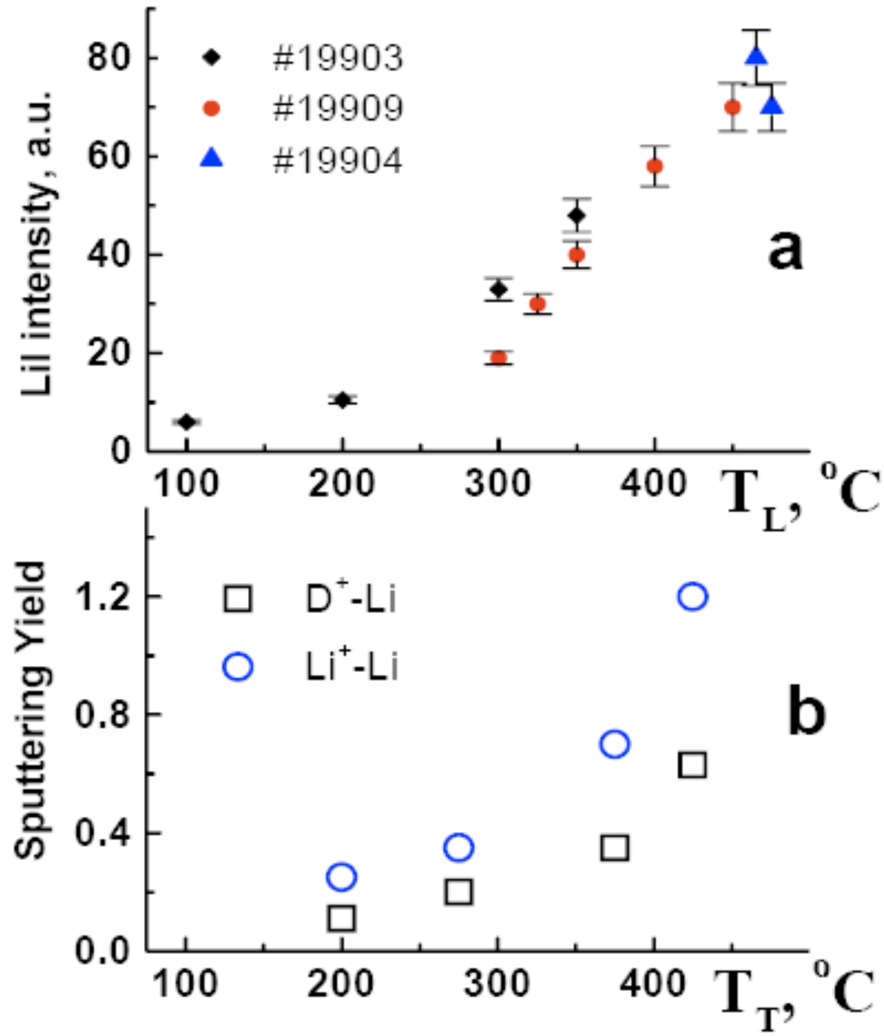




- D implanted at the lithium surface will lead to preferential sputtering of D atoms over Li leading to Li sputter yield reductions of ~ 40%[1]
- TDS measurements (Sugai, Baldwin, Evtikhin[2], Mirnov[3] and others) **show indirect evidence that D is implanted at the surface in solution with Li atoms based on their emission at temperatures (~ 400-500 C) lower than formation temp. for Li-D (T ~ 700 C)**

[1] J.P. Allain and D.N. Ruzic, Nucl. Fusion 42 (2002) 202.
[2] V.A. Evtikhin, et al. Plasma Phys. and Controlled Fusion, 44 (2002) 955.
[3] S. Mirnov, et al. J. Nucl. Mater. in press 2009

# *Enhancement in lithium erosion in T-11M*

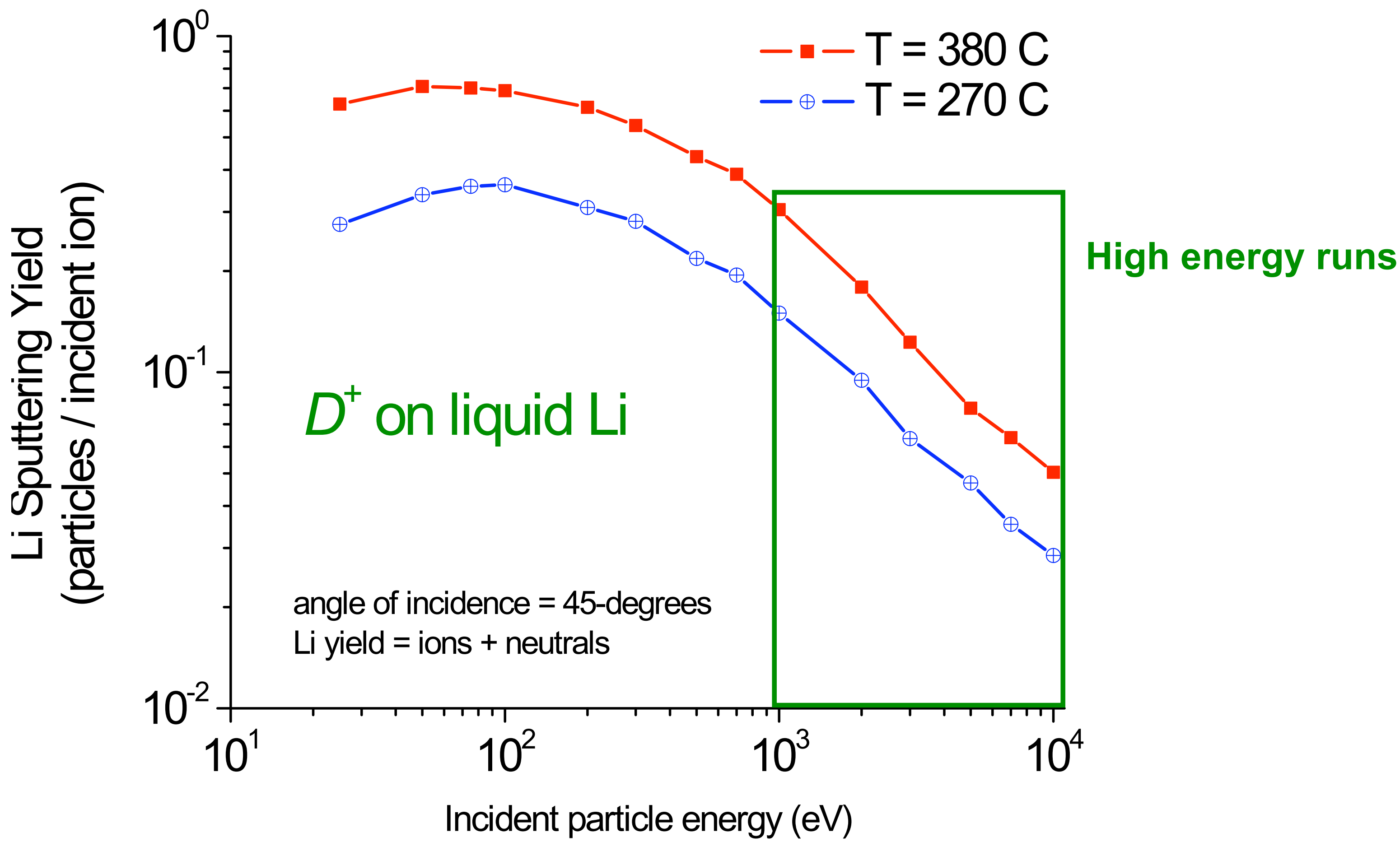
- Lithium capillary porous structure work in T-11M by Mirnov et al.[1]
- Particle-beam data shown in (b) is qualitatively consistent with lithium behavior with tokamak plasmas




[1] S. Mirnov, et al. Plasma Phys. Contr. Fus. 48 (2006) 821

RSSEL PURDUE ENGINEERING

# *Sputtering yields for liquid lithium at higher incident energies*



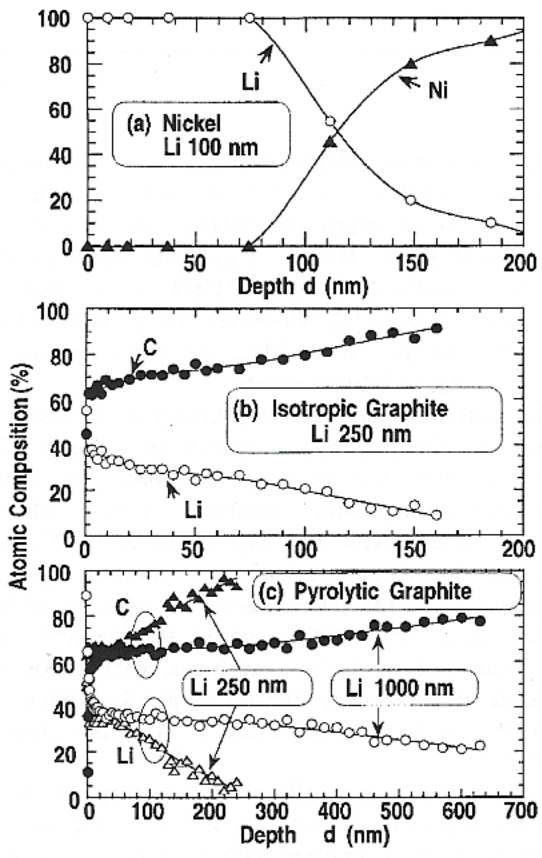
# *What have we learned about liquid-lithium surfaces exposed to energetic D, He and Li bombardment?*

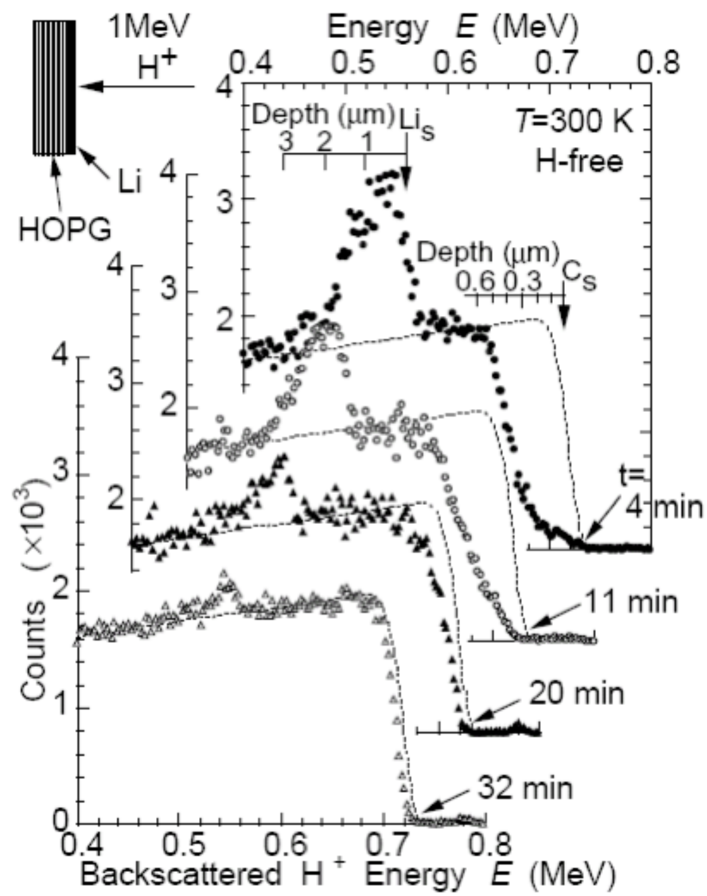
- No significant difference in sputtering from the solid to liquid state of lithium when temperature is near melting point
- Non-linear increase in sputtering from liquid-Li when temperature is about 50% higher than melting point (accounting for evaporation)
- Two-thirds of lithium sputtered particles are in the charged state
- Implanted hydrogen leads to a ~ 40% decrease in *lithium* sputtering
- So far: liquid Li, Sn-Li, Ga and Sn show signs of erosion enhancement (particularly lithium) *with* rise in temperature
- Li-DiMES data shows near-surface ionization of emitted Li particles within ~ 1cm[1]
- High retention of deuterium in liquid lithium (PISCES-B results by M. Baldwin et al.)[2]

[1] J.P. Allain J.N. Brooks, and D.G. Whyte, Nucl Fusion, 44 (2004) 655.

[2] M. Baldwin, R.P. Doerner, R. Causey, et al. J. Nucl. Mater. 306 (2002) 15

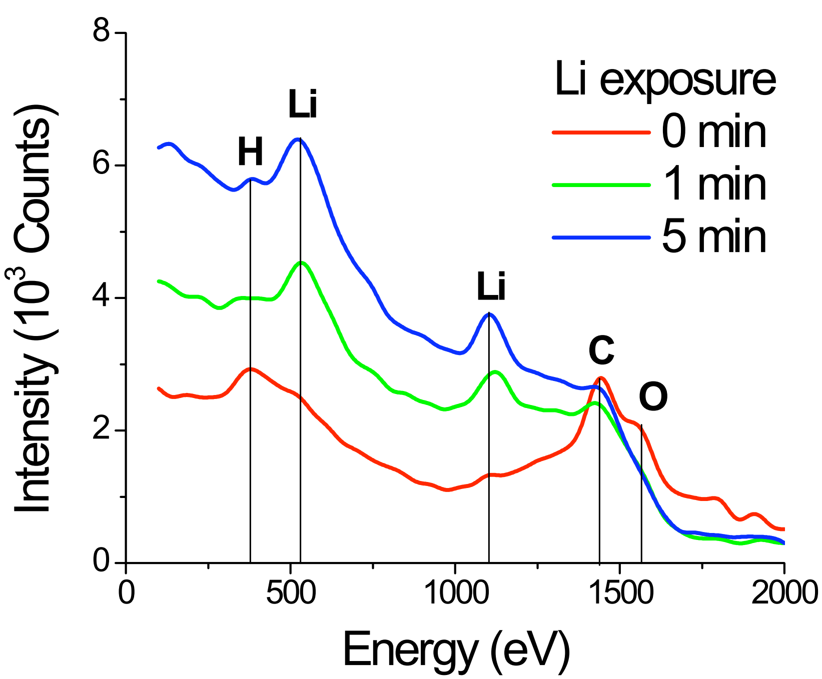
# *H. Sugai's work on lithium intercalation in graphite*

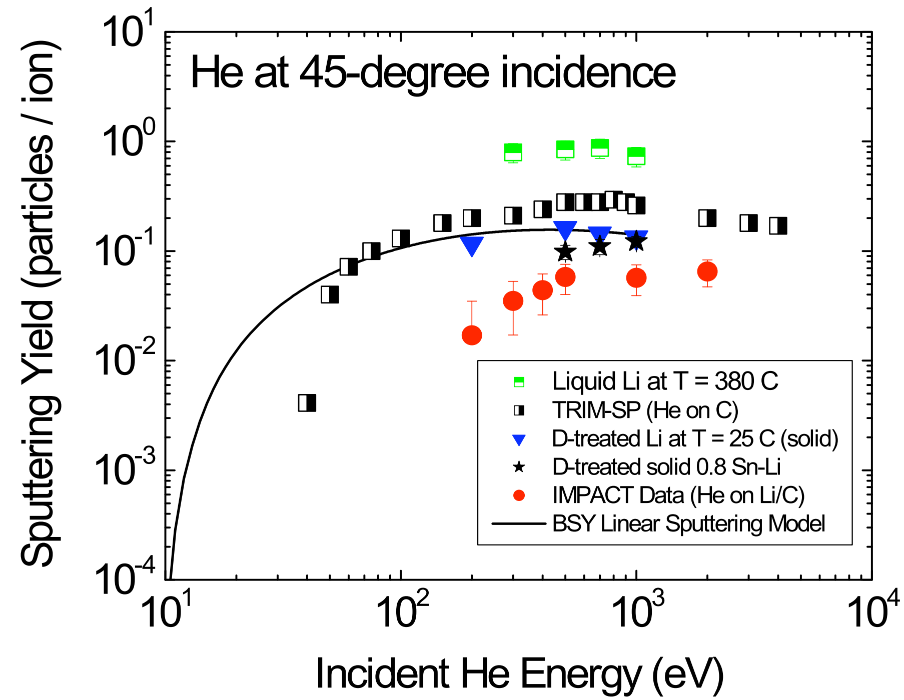



N. Itou, H. Toyoda, K. Morita, H. Sugai, J. Nucl. Mater. 290-293 (2001) 281.

# *Lithium coatings on graphite: surface effects on erosion, particle retention*



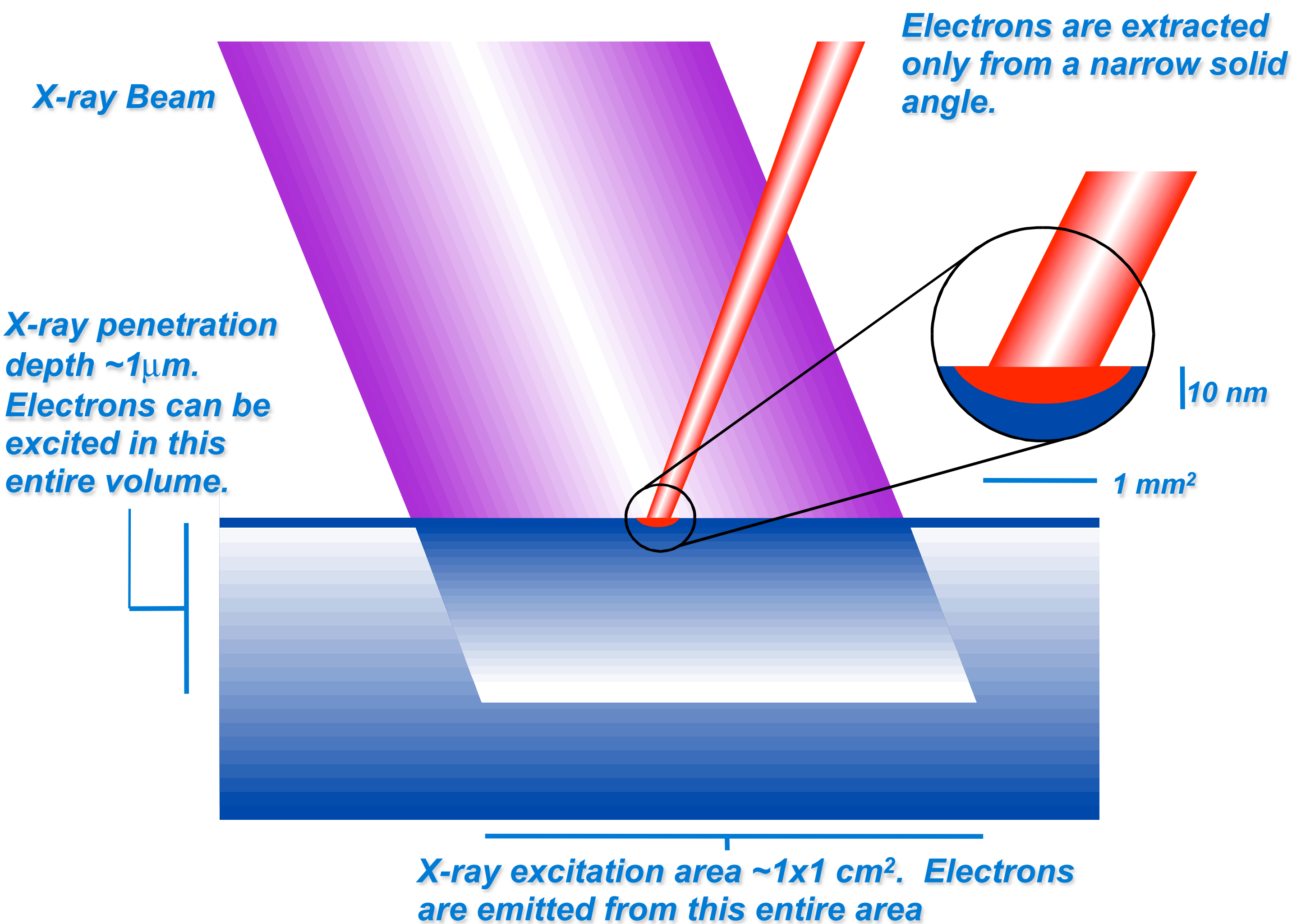




- *Nominally* lithium intercalates to the basal planes of graphite. Difficult to maintain 100% lithium layers on top few ML. Oxygen typically bound with lithium
- Substantial reduction of both *physical* and *chemical* sputtering by *D or He* bombardment when comparing lithiated graphite surfaces to either pure Li or C

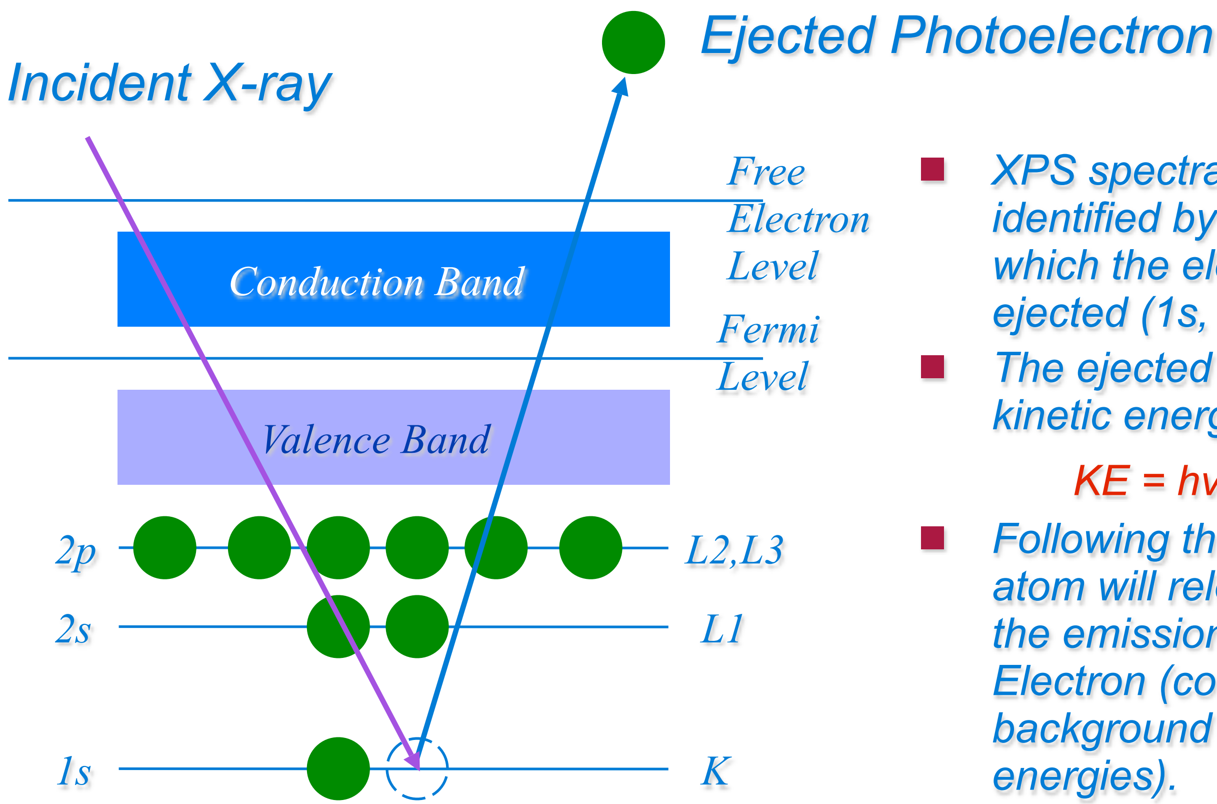
*J.P. Allain, D.L. Rokusek, et al. J. Nucl. Mat. 390-391 (2009) 942*

# X-ray Photoelectron Spectroscopy

## Small Area Detection



# The Photoelectric Process




- XPS spectral lines are identified by the shell from which the electron was ejected (1s, 2s, 2p, etc.).
- The ejected photoelectron has kinetic energy:

$$KE = hv - BE - \Phi$$

- Following this process, the atom will release energy by the emission of an Auger Electron (contributes to background at high binding energies).

# XPS Energy Scale- Binding energy

$$BE = h\nu - KE - \Phi_{spec}$$

*Where:* *BE= Electron Binding Energy*

*KE= Electron Kinetic Energy*

*$\Phi_{spec}$= Spectrometer Work Function*

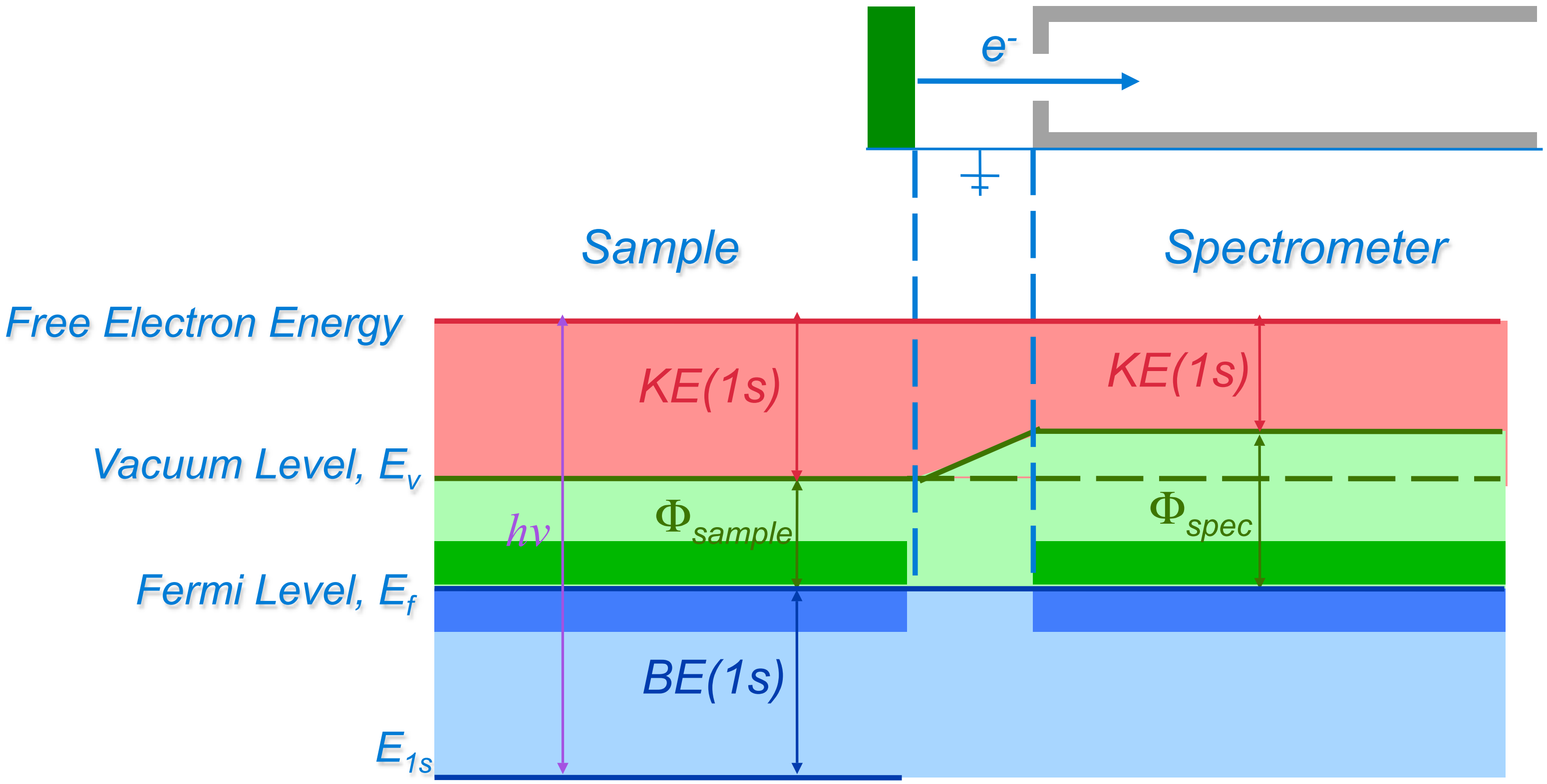
*Photoelectron line energies: Not Dependent on photon energy.*

*Auger electron line energies: Dependent on photon energy.*

*The binding energy scale was derived to make uniform comparisons of chemical states straight forward.*

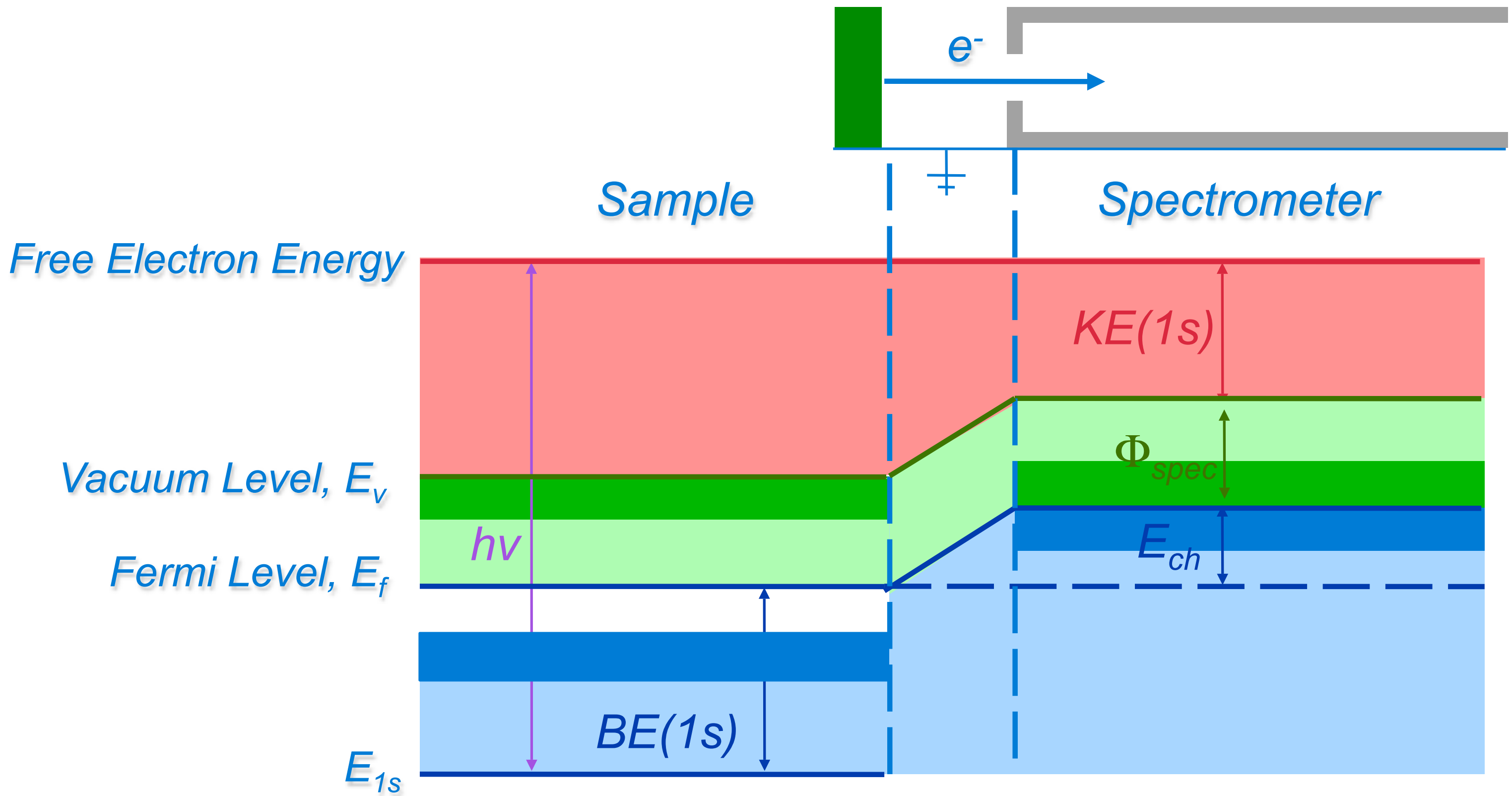
*Chemical states of either "binding" between atoms (C-O) or "functional" behavior (functionalities) between atoms (e.g. dipole/induced-dipole interactions between H in C structures due to Li electron transfer)*

# Sample/Spectrometer Energy Level Diagram- Conducting Sample




*Because the Fermi levels of the sample and spectrometer are aligned, we only need to know the spectrometer work function, $\Phi_{spec}$, to calculate BE(1s).*

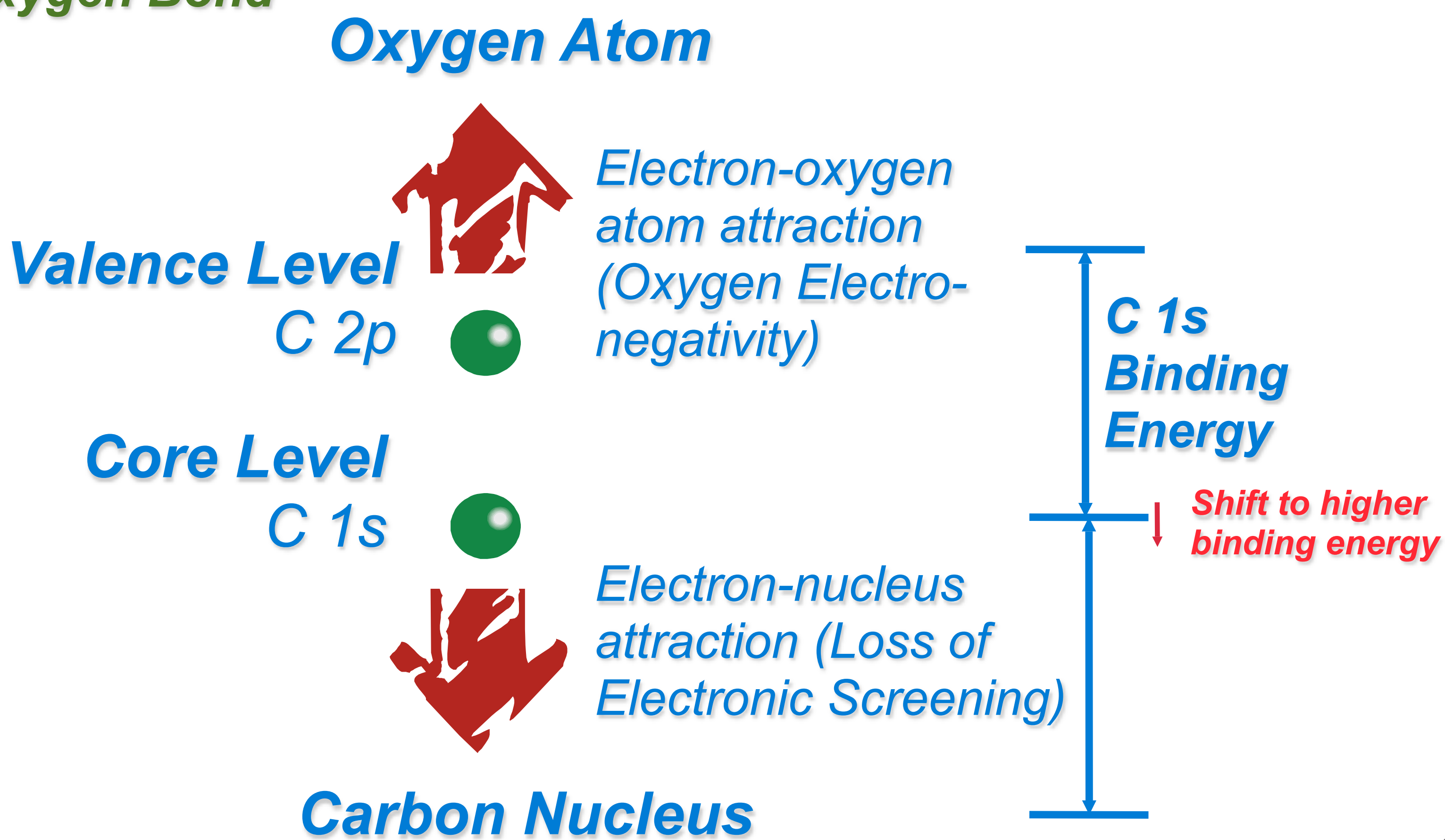
# Sample/Spectrometer Energy Level Diagram- Insulating Sample

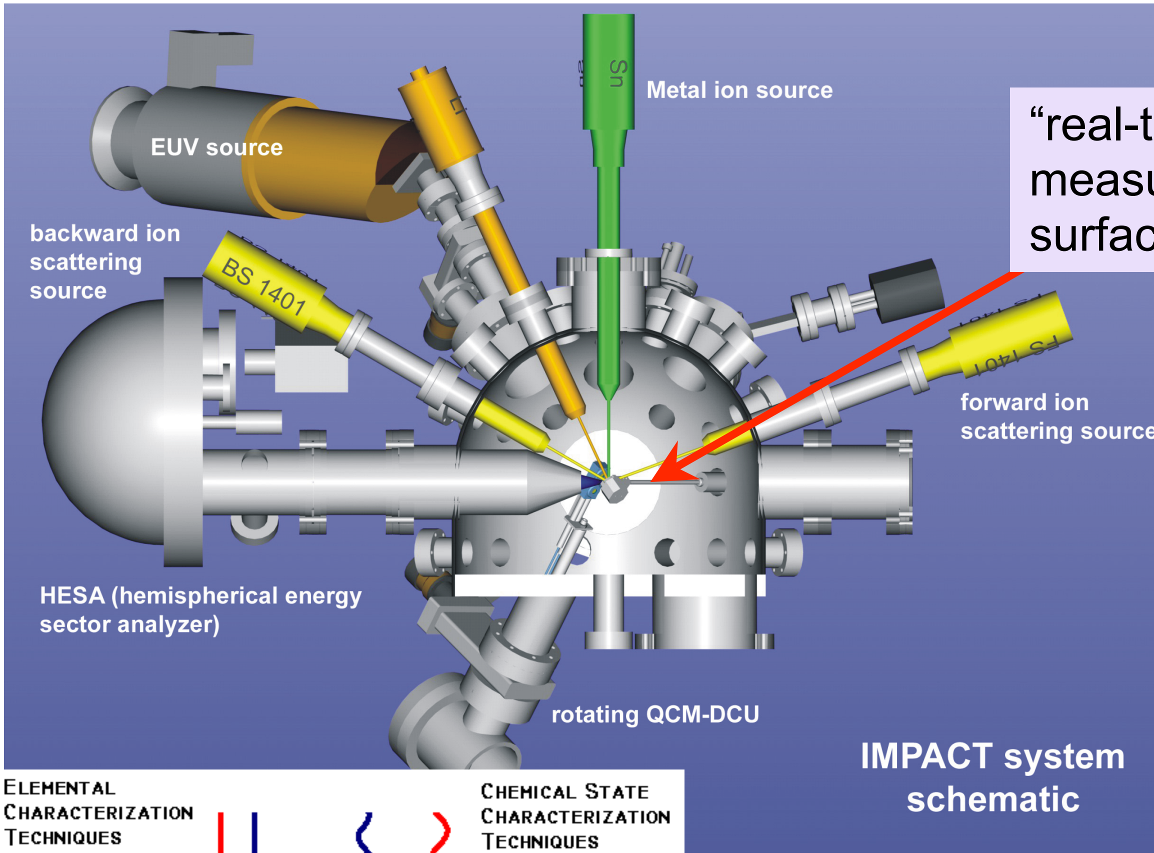



A relative build-up of electrons at the spectrometer raises the Fermi level of the spectrometer relative to the sample. A potential $E_{ch}$ will develop.

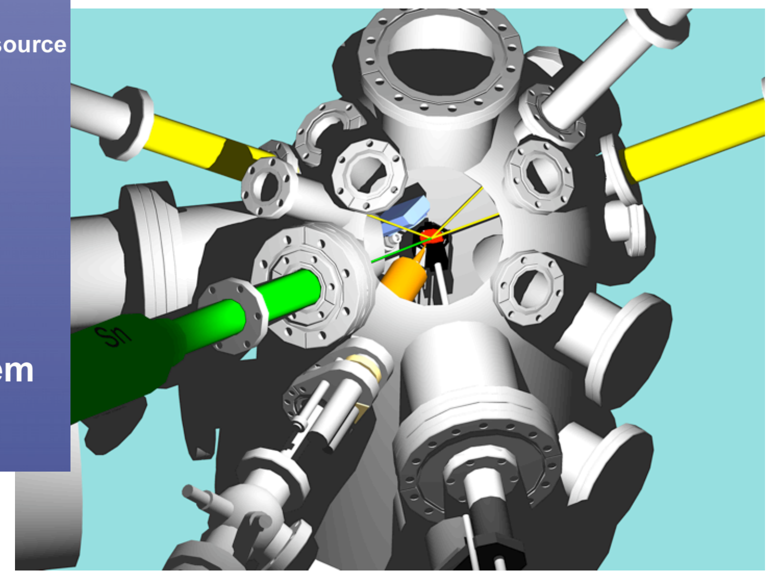
# Chemical Shifts-
# Electronegativity Effects

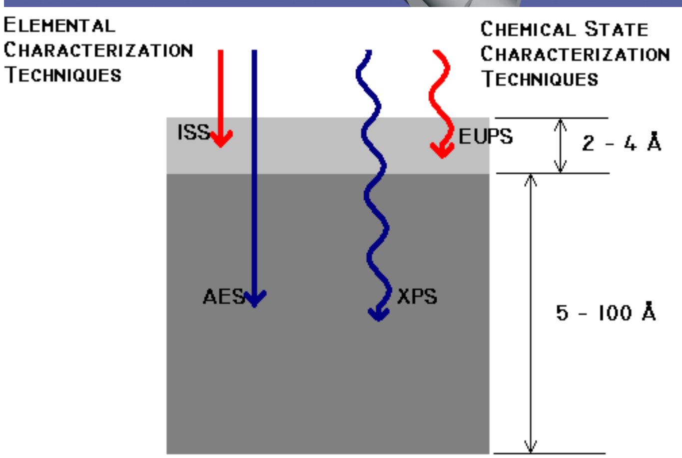
**Carbon-Oxygen Bond**





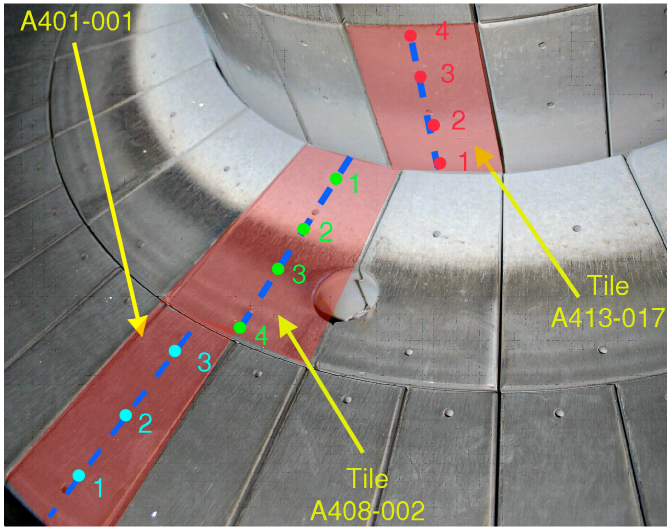

“real-time” erosion rate measurement *during* analysis from surface with QCM-DCU system

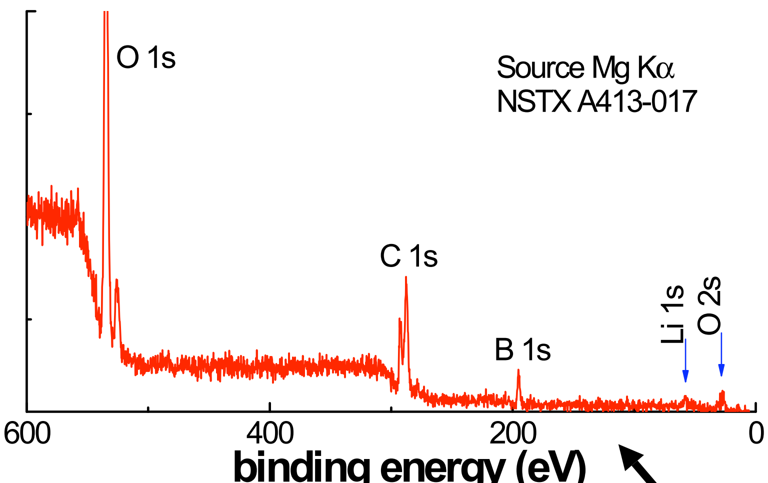


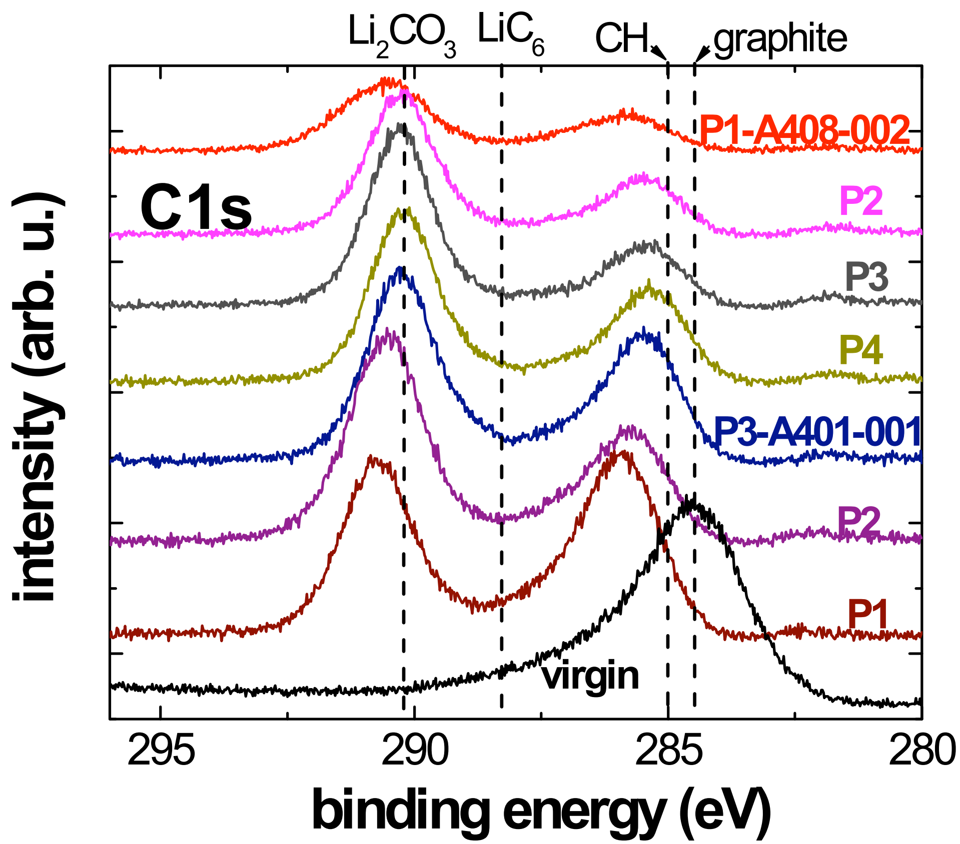



***IMPACT*** and ***PRIHSM*** use several electron spectroscopies for surface chemical analysis: XPS, UPS and ARPES with ion scattering spectroscopies: forward and backward scattering modes

# NSTX tiles showed presence of $Li_2CO_3$



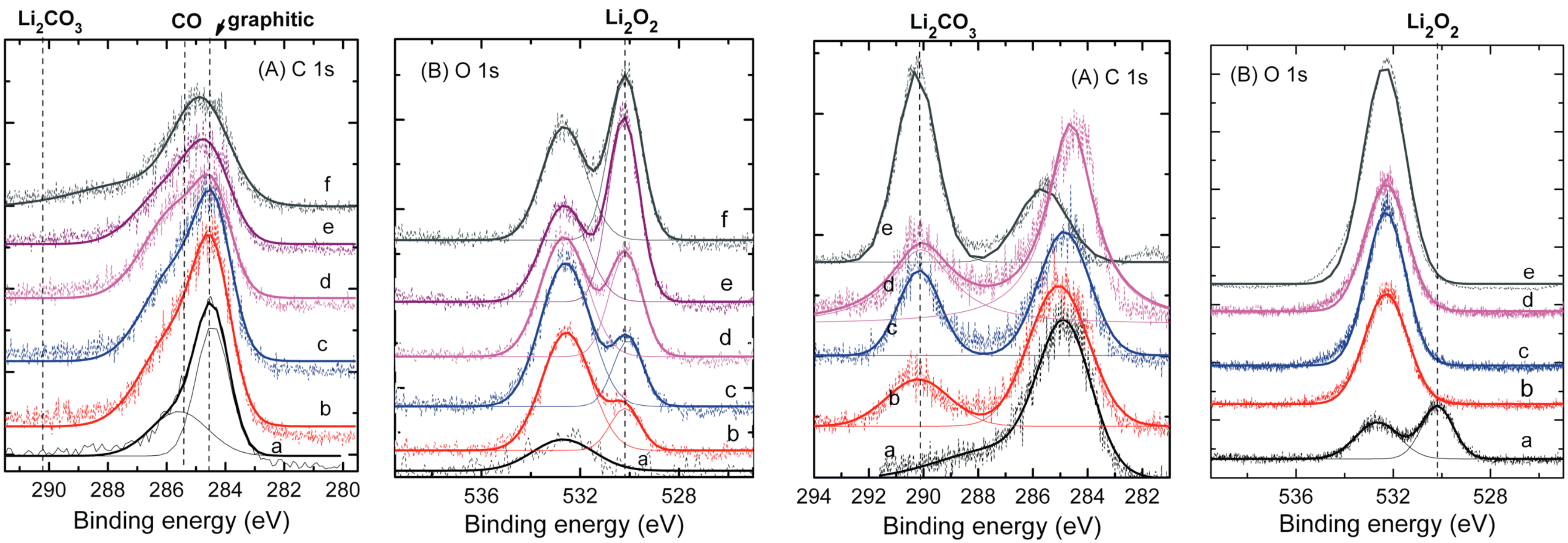




XPS spectra of NSTX tiles show presence of carbonate

J.P. Allain et al. *J. Nucl. Mater. 390-391 (2009) 942*

Typical XPS spectrum from a NSTX Li-coated ATJ graphite tile

# *Controlled in-situ lithium deposition on ATJ graphite followed by air exposure*




C1s Li deposition O1s C1s Exposure to air O1s

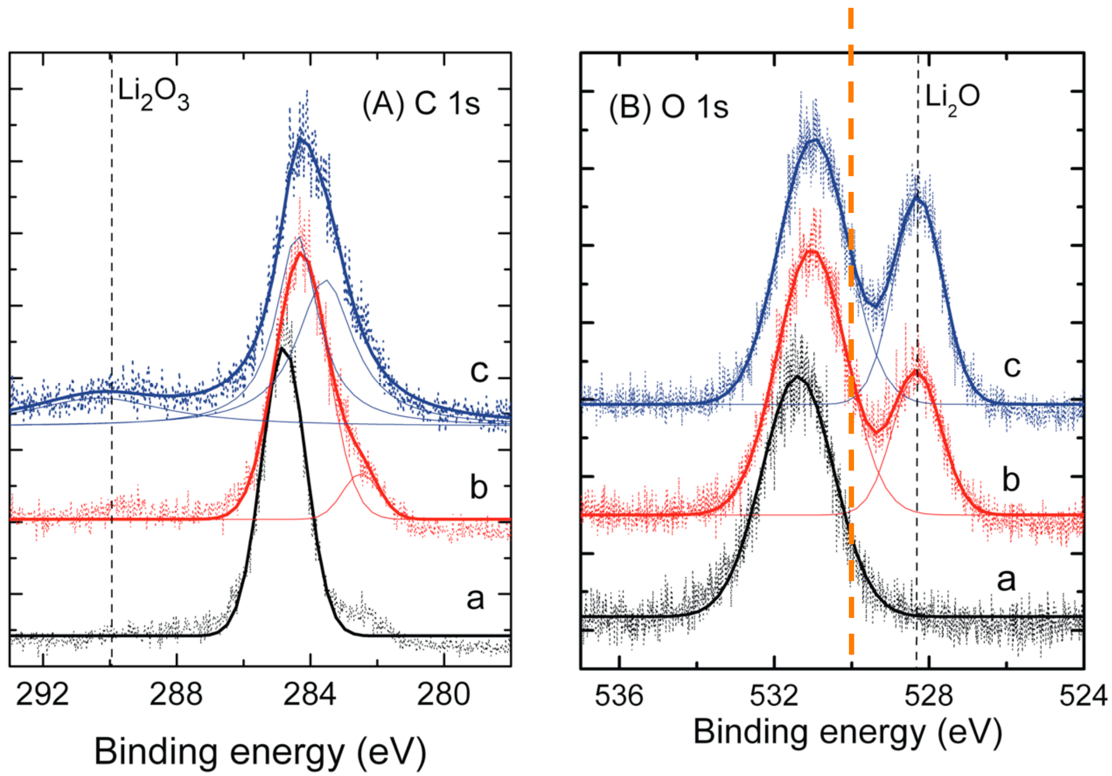
- Lithium deposition yields peak at 529.5 eV ± 0.5 eV
- Exposure to air yields peak at 290 eV and 529 eV peak disappears

RSSEL

PURDUE ENGINEERING

# *Control group with "pure lithium" target*

- Pure 'as received' Li is used and transferred to the chamber in an Ar environment
- Strong sample charging effects observed, graphitic bond at 284.5eV is used for calibration
- O1s peak appears at 531.5 eV which corresponds to $Li_2CO_3$ and/ or LiOH
- $Li_2CO_3$ peak at 290.2 eV is found to be weak in the XPS spectra of C1s.
- After $Ne^+$ etching a strong O1s peak appeared at 528.5 eV and assigned to $Li_2O$
- The peroxide (529eV ± 0.5 eV ) functionality is not observed on Li metal

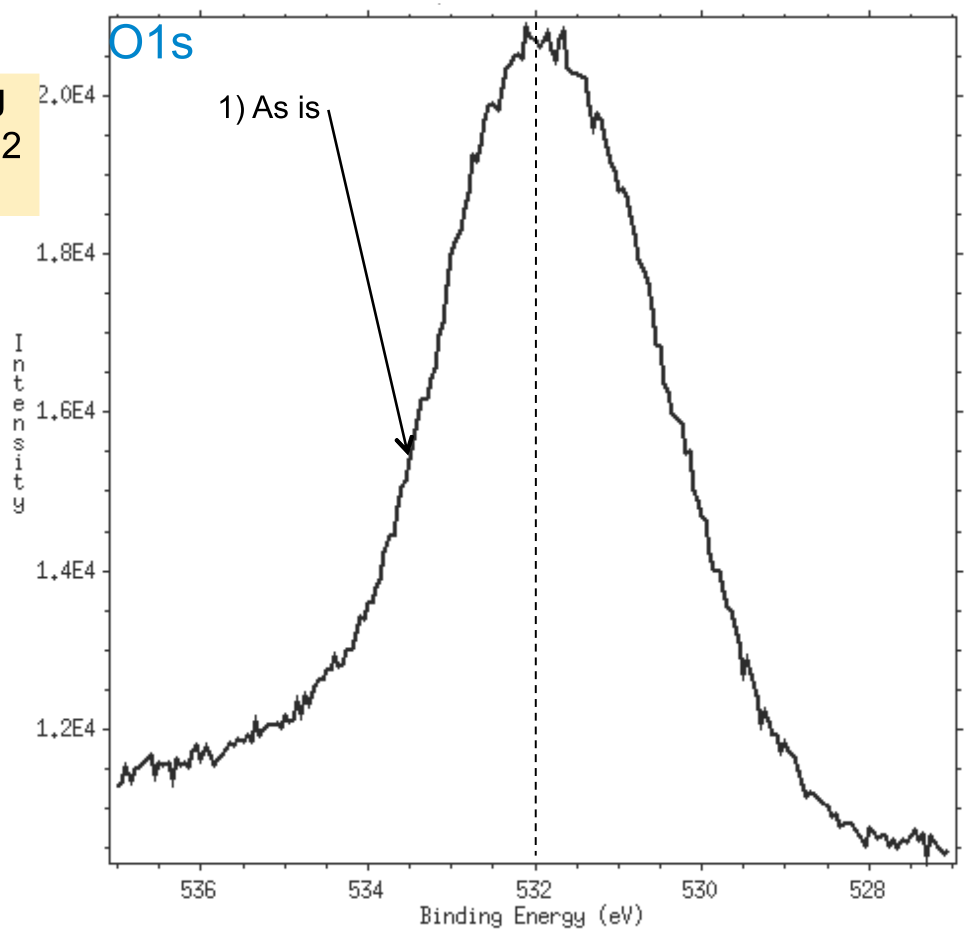



From "a" to "c" removal of surface oxide layer


J.P. Allain, D. Rokusek, et al., J. Nucl. Mater. 390-391 (2009) 942
S.S. Harilal and J.P. Allain, Appl. Surface Sci. in press 2009

# *Results – Li-D-O functionality*

1) The O1s peak on ATJ graphite is located at 532 eV

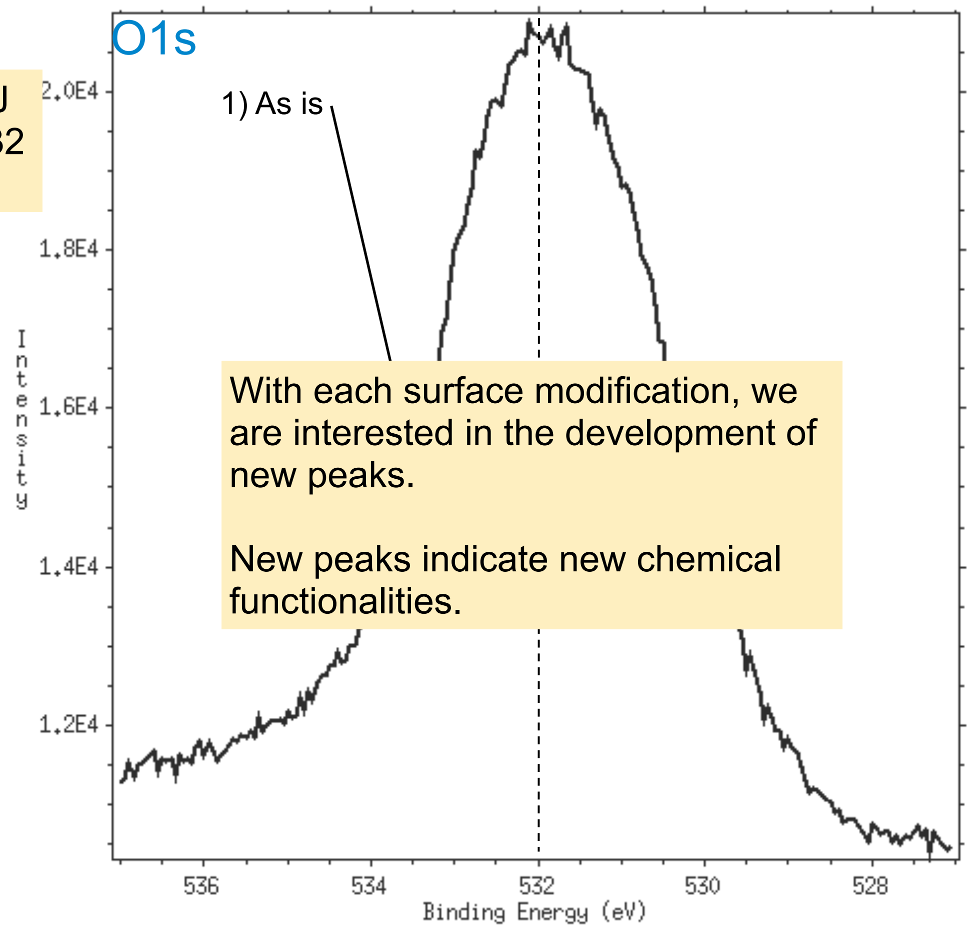





PURDUE ENGINEERING

# Results – Li-D-O functionality

1) The O1s peak on ATJ graphite is located at 532 eV




With each surface modification, we are interested in the development of new peaks.

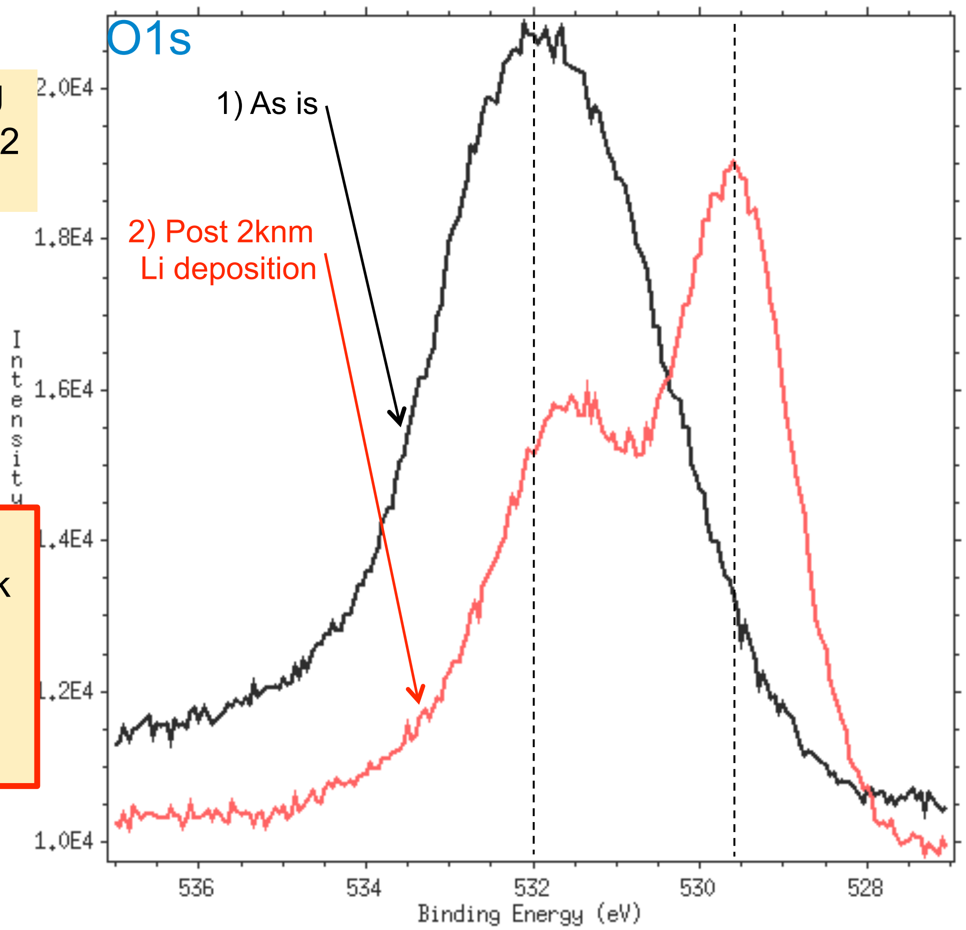
New peaks indicate new chemical functionalities.



PURDUE ENGINEERING

# Results – Li-D-O functionality

1) The O1s peak on ATJ graphite is located at 532 eV

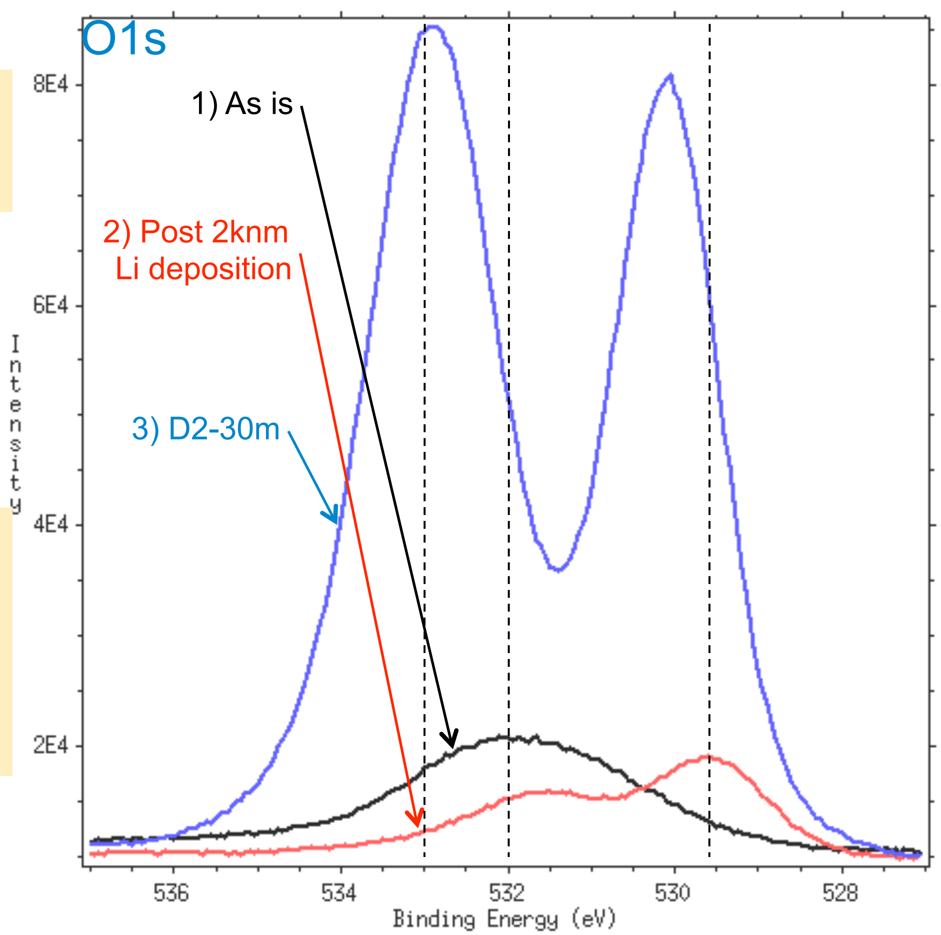



2) Lithium deposition results in a second peak at ~529.5 eV. A slight shift to lower binding energy in the 532 eV also occurs.

# Results – Li-D-O functionality

1) The O1s peak on ATJ graphite is located at 532 eV

2) Lithium deposition results in a new second peak at ~529.5 ± .5 eV. A slight shift to lower binding energy in the 532 eV also occurs.




3) 30 minute deuterium irradiation (Γ ≈ 1.5 E15 $cm^{-2}$) causes a new peak to develop at 533 eV, and a slight shift to higher binding energy for the 529.5 eV peak.

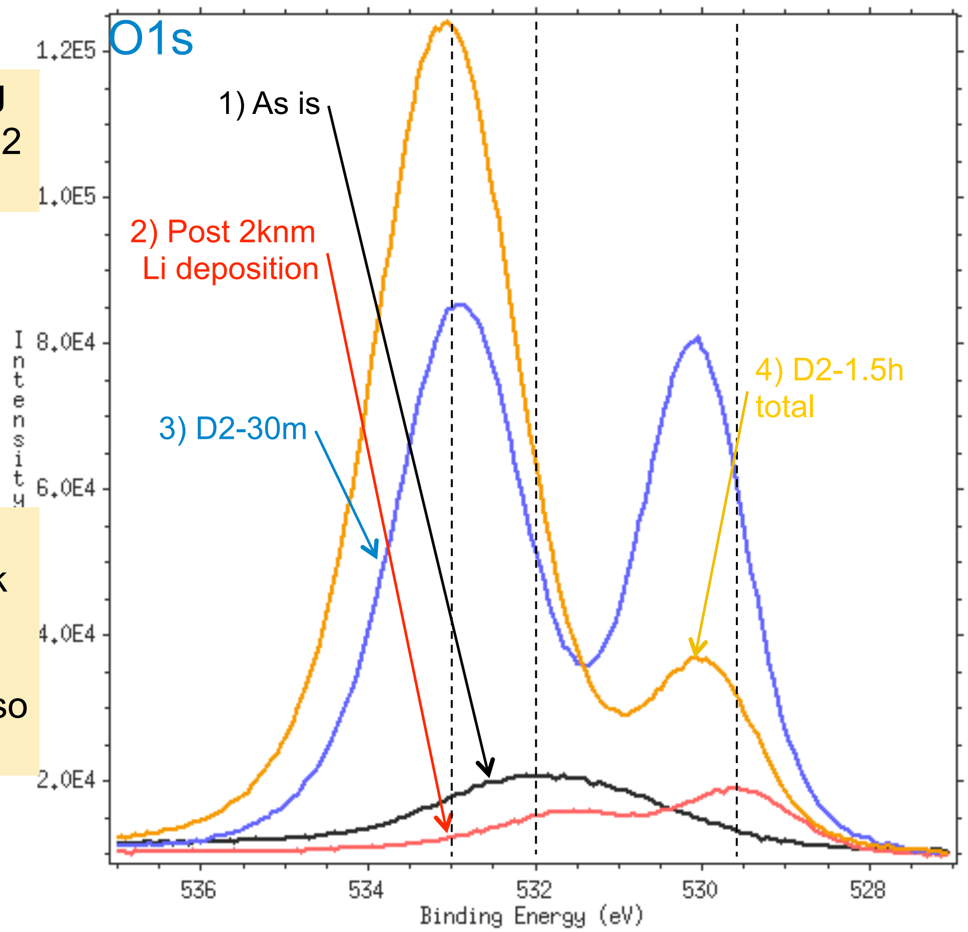


PURDUE ENGINEERING

# Results – Li-D-O functionality

1) The O1s peak on ATJ graphite is located at 532 eV

2) Lithium deposition results in a second peak at ~529.5 eV. A slight shift to lower binding energy in the 532 eV also occurs.



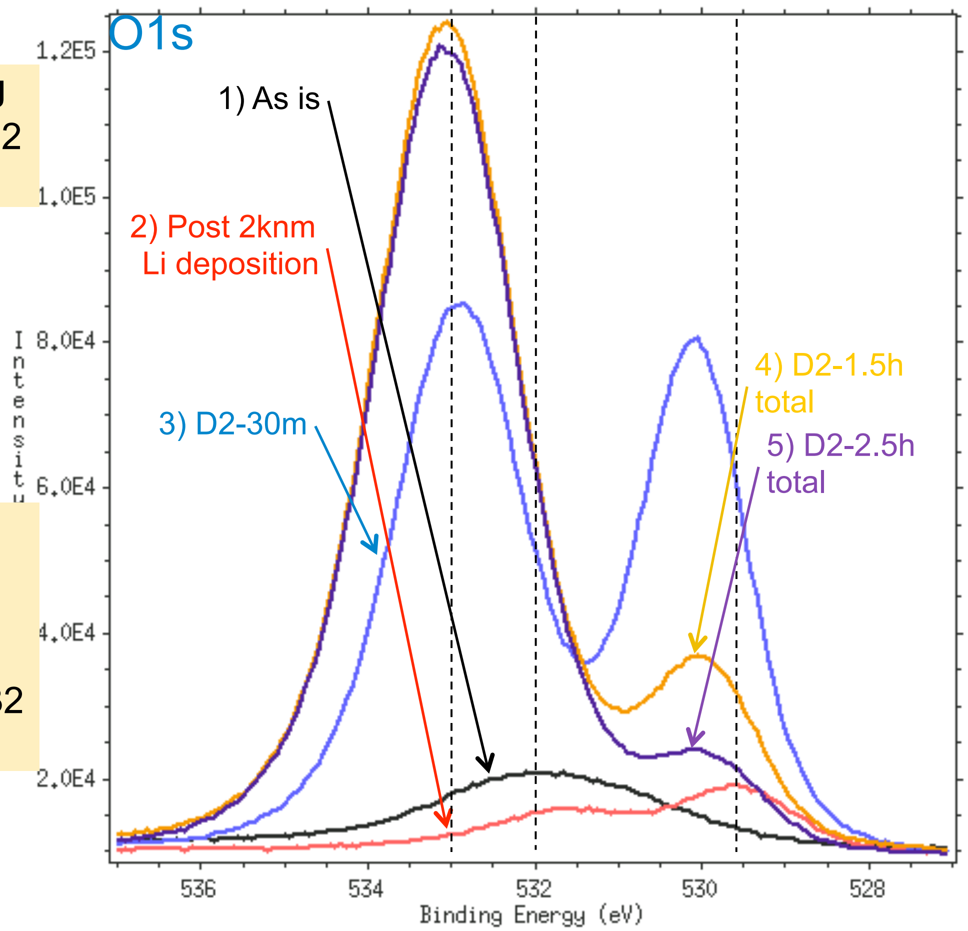

3) 30 minute deuterium irradiation ($\Gamma \approx 1.5$ E15 $cm^{-2}$) causes a new peak to develop at 533 eV, and a slight shift to higher binding energy for the 529.5 eV peak.

4) The relative intensity of the 533 eV peak compared to the 529.5 eV peak increases with subsequent irradiations.

# *Results – Li-D-O functionality*

1) The O1s peak on ATJ graphite is located at 532 eV

2) Lithium deposition results in a new second peak at ~529.5 ± .5 eV. A slight shift to lower binding energy in the 532 eV also occurs.




3) 30 minute deuterium irradiation (Γ ≈ 1.5 E15 $cm^{-2}$) causes a new peak to develop at 533 eV, and a slight shift to higher binding energy for the 529.5 eV peak.

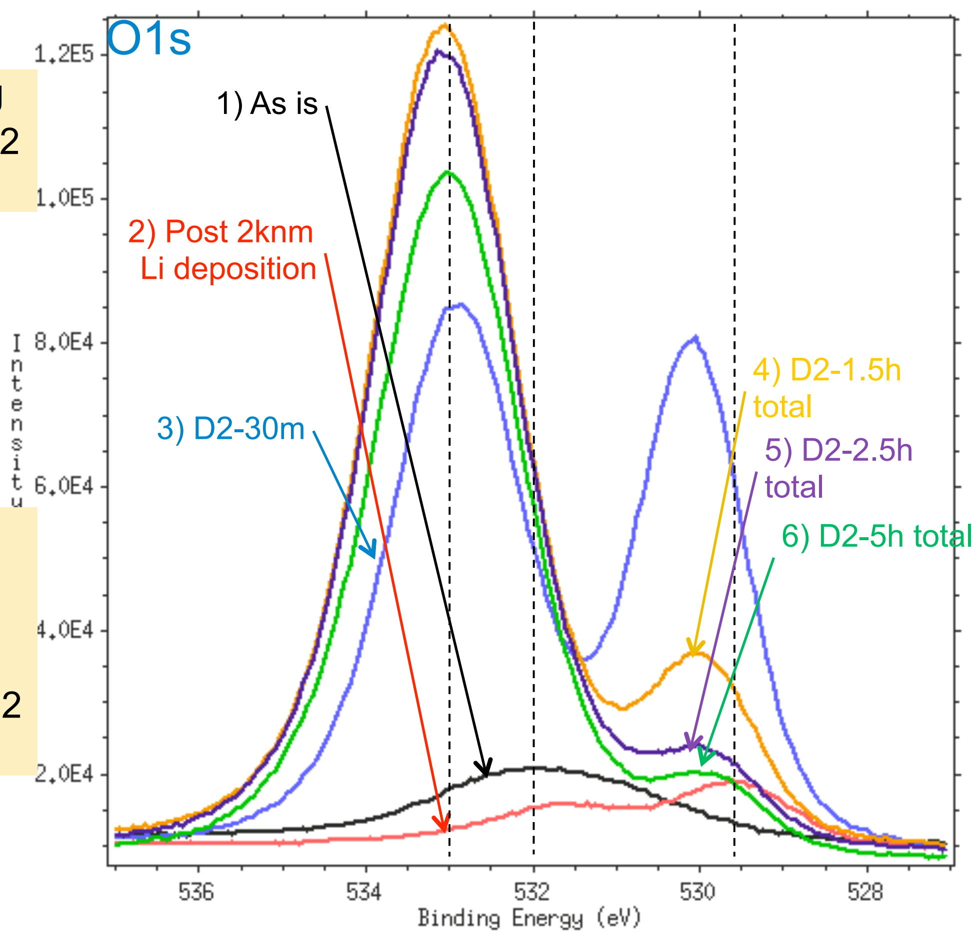
4) The relative intensity of the 533 eV peak compared to the 529.5 eV peak increases with subsequent irradiations.

5) …and again...

# Results – Li-D-O functionality

1) The O1s peak on ATJ graphite is located at 532 eV

2) Lithium deposition results in a new second peak at ~529.5 ± .5 eV. A slight shift to lower binding energy in the 532 eV also occurs.




3) 30 minute deuterium irradiation ($\Gamma \approx 1.5$ E15 $cm^{-2}$) causes a new peak to develop at 533 eV, and a slight shift to higher binding energy for the 529.5 eV peak.

4) The relative intensity of the 533 eV peak compared to the 529.5 eV peak increases with subsequent irradiations.

5) …and again...

6) …and again.

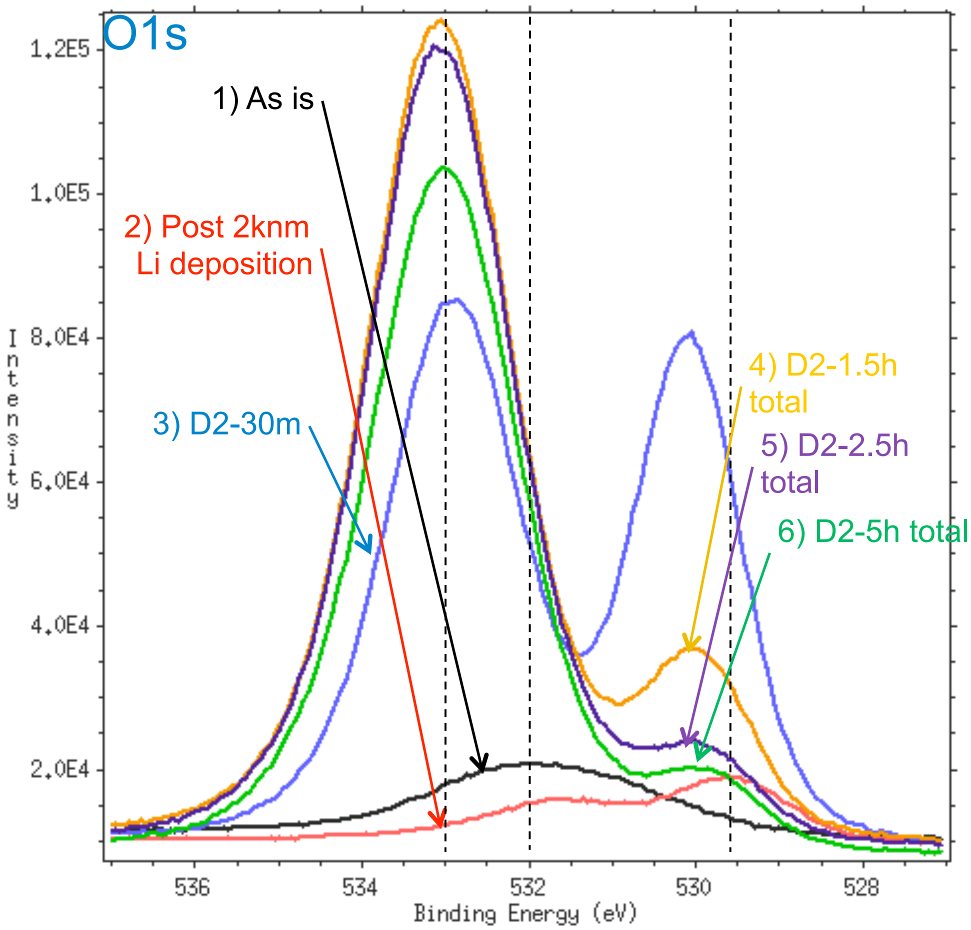


PURDUE ENGINEERING

# Results – Li-D-O functionality

## Observations

Based on these results and control experiments



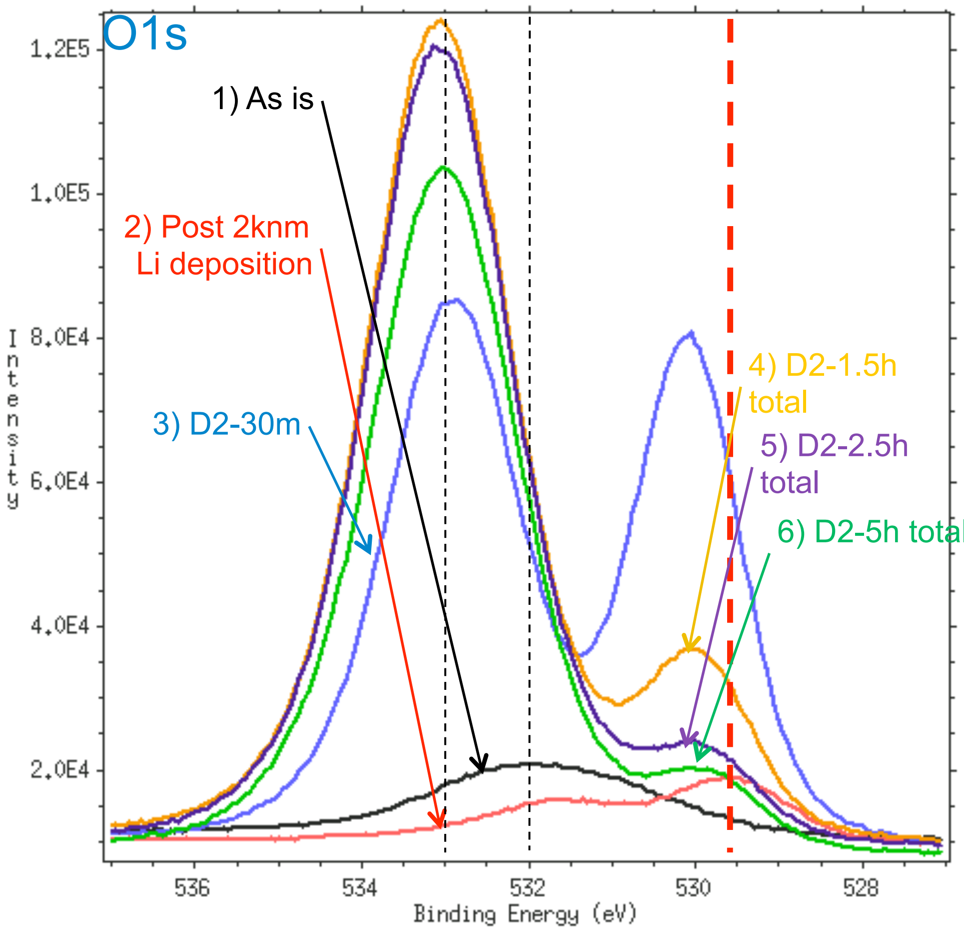



PURDUE ENGINEERING

# *Results – Li-D-O functionality*

## Observations

Based on these results and control experiments




**529 eV**

- Only develops after Li deposition
- Shifts slightly (~.5 eV) after D2.
- Relative intensity decreases with higher D2 fuence



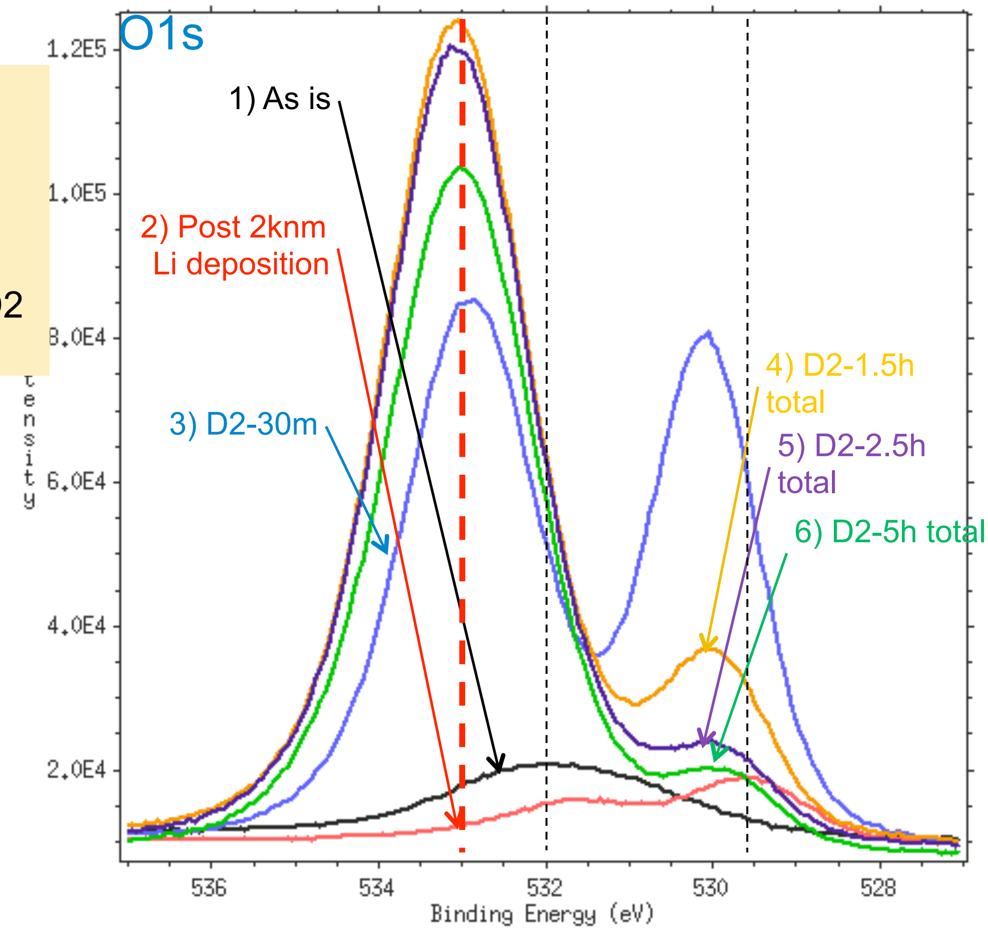
PURDUE ENGINEERING

# Results – Li-D-O functionality

**Observations**

Based on these results and control experiments

**533 eV**
- Only develops after irradiating a *lithiated* sample.
- Relative intensity increases with higher D2 fluence




**529 eV**
- Only develops after Li deposition
- Shifts slightly (~.5 eV) after D2.
- Relative intensity decreases with higher D2 fuence

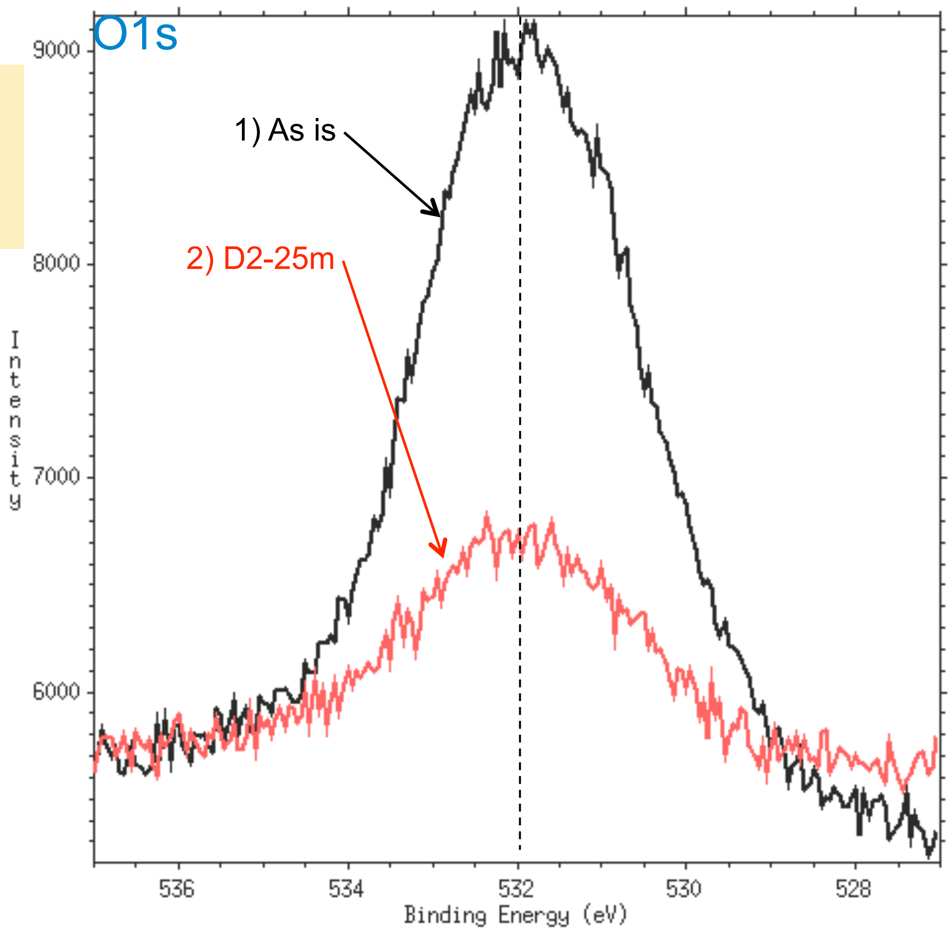


PURDUE ENGINEERING

# Results – Li-D-O functionality

Control experiment

**Procedure:**
ATJ graphite was irradiated with D *without* any lithium conditioning.

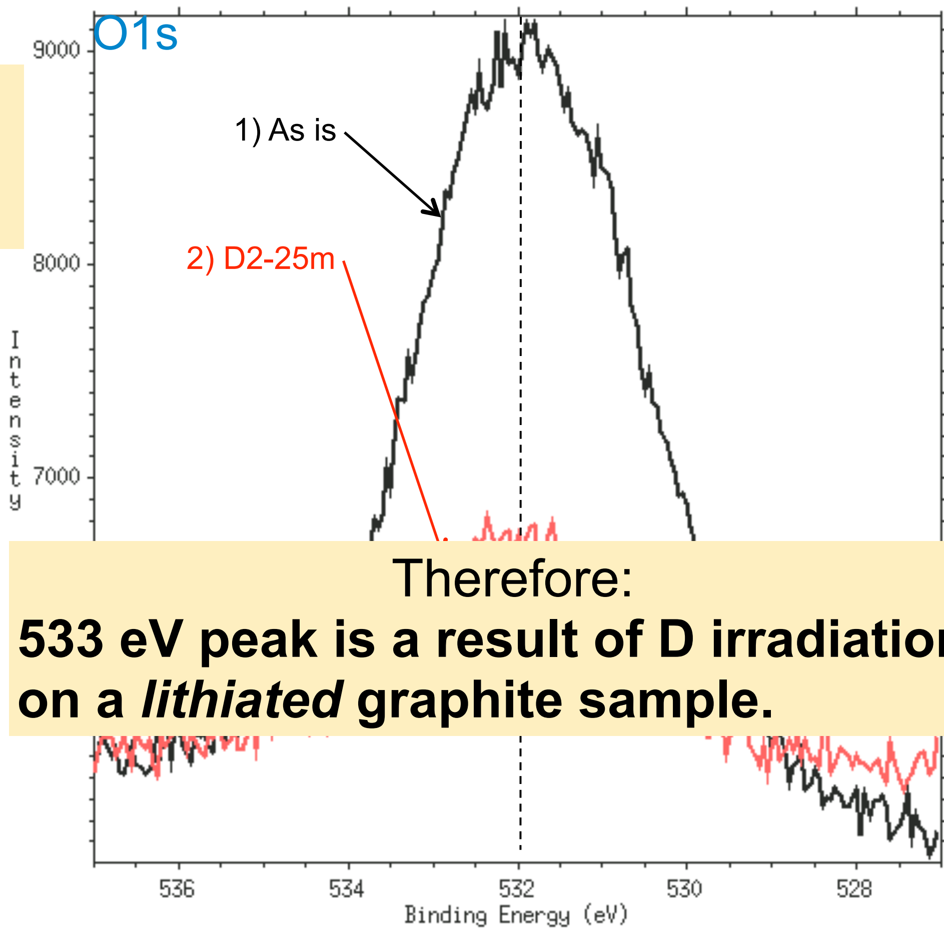



**Result:**
No shifts or new peaks were observed.

# Results – Li-D-O functionality

Control experiment

**Procedure:**
ATJ graphite was irradiated with D *without* any lithium conditioning.

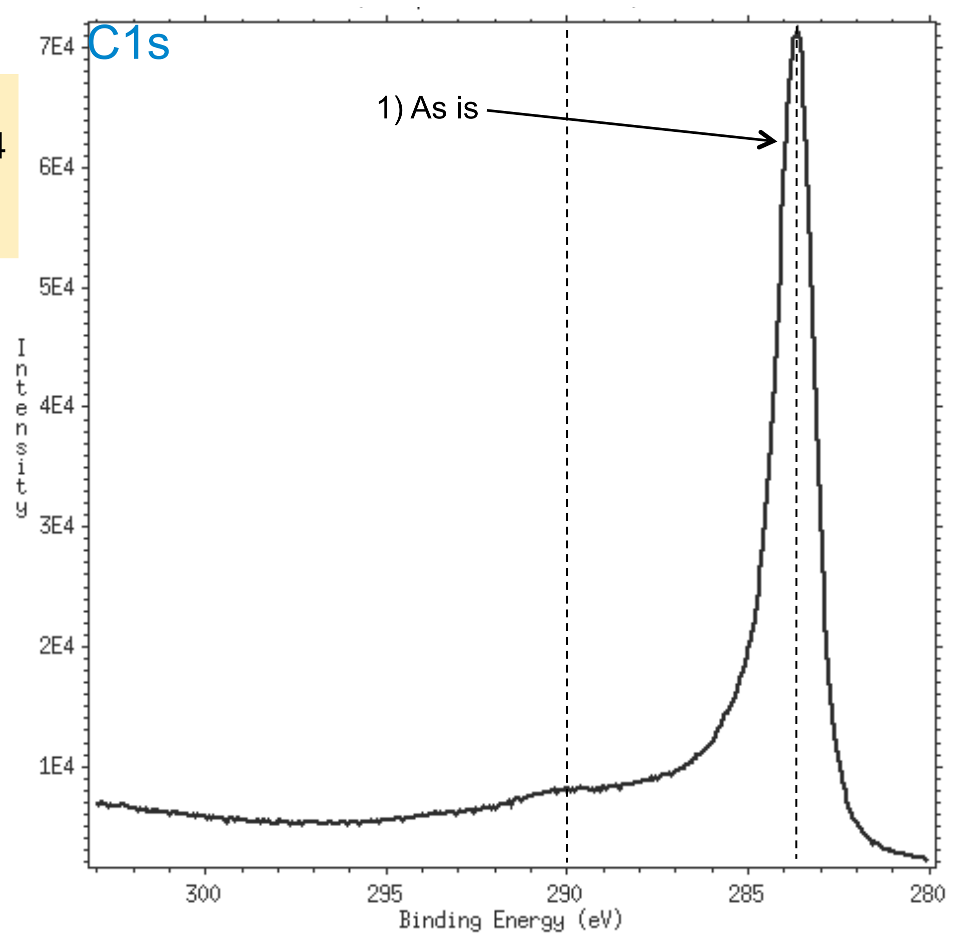



**Result:**
No shifts or new peaks were observed.

Therefore:
**533 eV peak is a result of D irradiation on a *lithiated* graphite sample.**

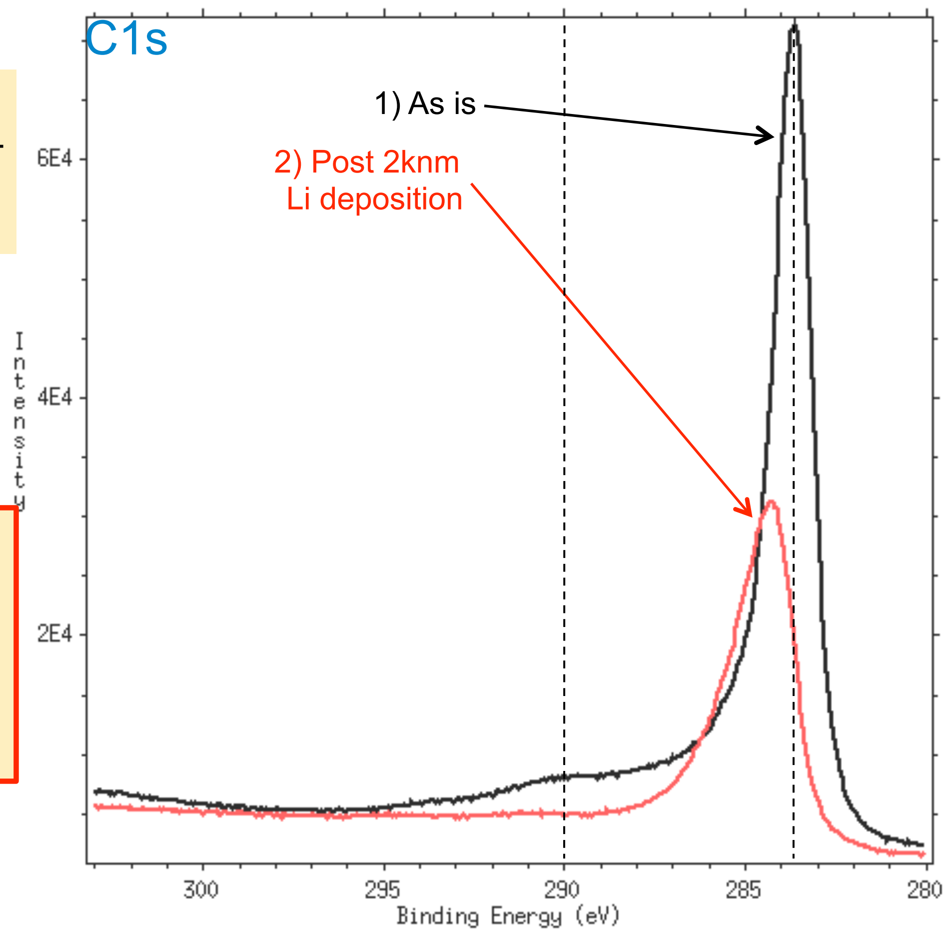
# Results – Li-D-O and C functionality

1) ATJ graphite shows a graphitic C1s peak at 284 eV. Carbonate presence is observed at 290 eV.



# *Results – Li-D-O and C functionality*

1) ATJ graphite shows a graphitic C1s peak at 284 eV. Carbonate presence is observed at 290 eV.

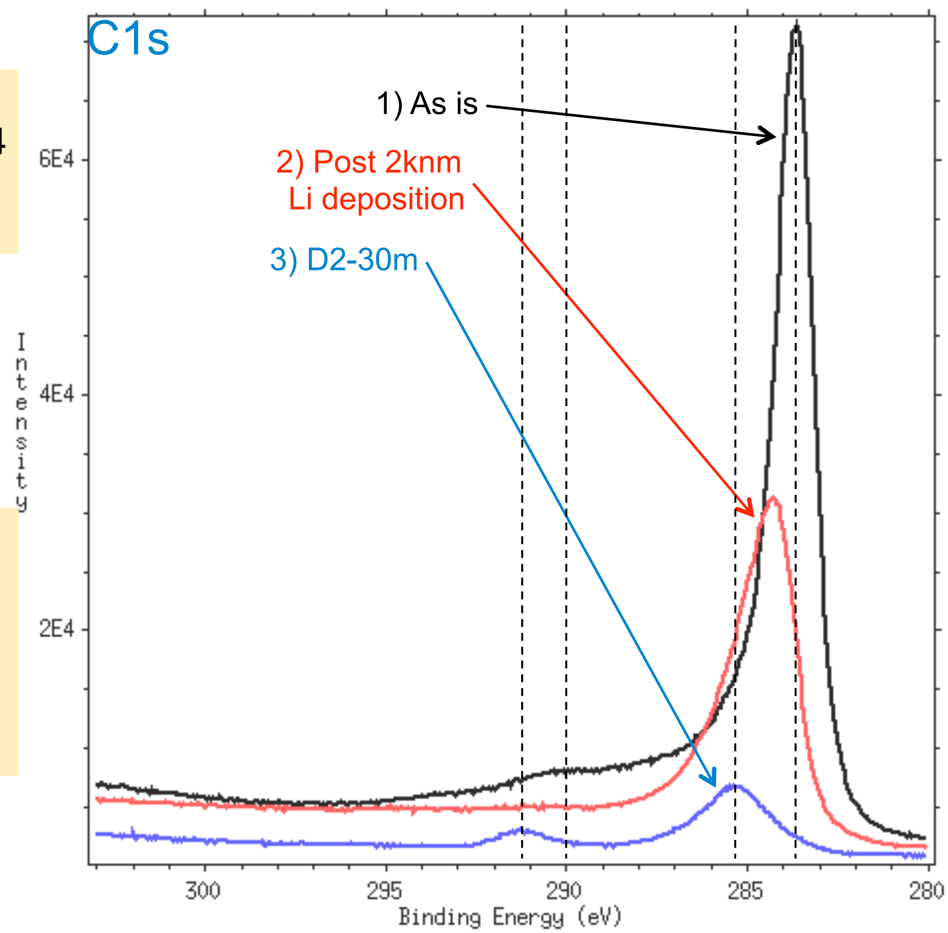



2) Lithium deposition results causes the FWHM of the primary peak to increase. Peak shifts ~1eV to higher binding energy.

# Results – Li-D-O and C functionality

1) ATJ graphite shows a graphitic C1s peak at 284 eV. Carbonate presence is observed at 290 eV.

2) Lithium deposition results causes the FWHM of the primary peak to increase. Peak shifts ~1eV to higher binding energy.




3) 30 minute deuterium irradiation (Γ ≈ 1.5 E15 $cm^{-2}$) causes a new peak to develop at 291 eV. The 284 eV peak shifts again to higher binding energy, now residing ~285 eV.

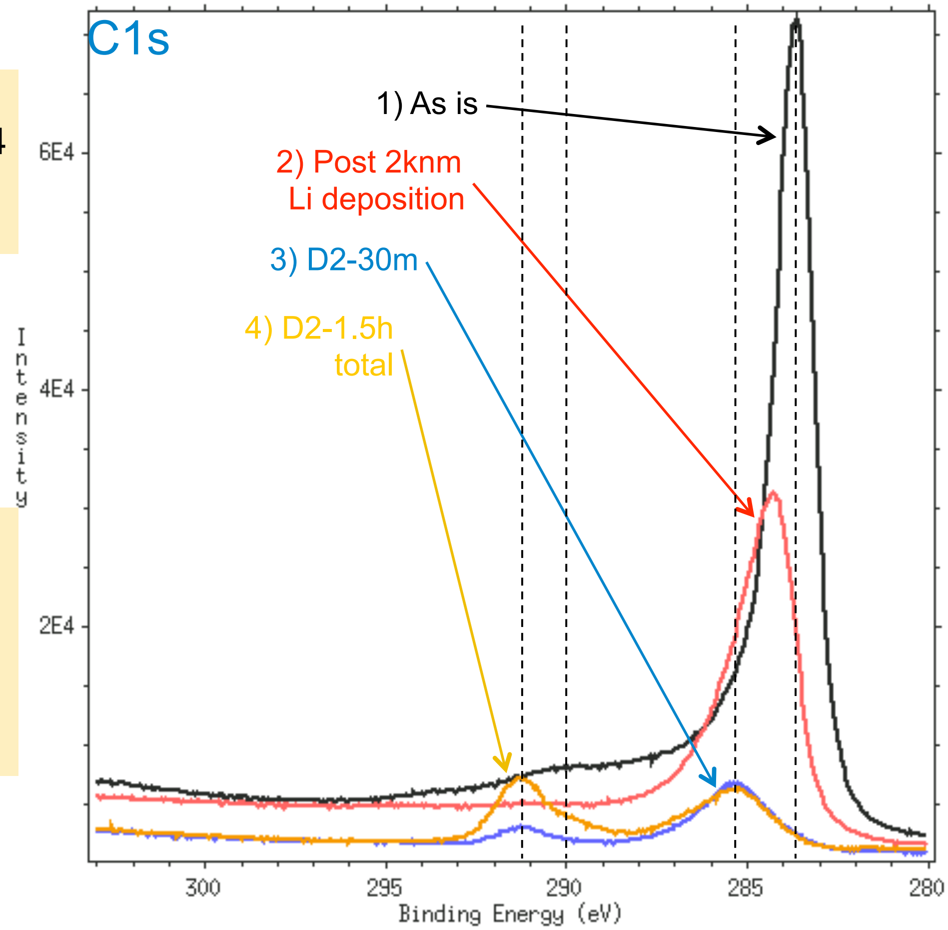


PURDUE ENGINEERING

# Results – Li-D-O and C functionality

1) ATJ graphite shows a graphitic C1s peak at 284 eV. Carbonate presence is observed at 290 eV.

2) Lithium deposition results causes the FWHM of the primary peak to increase. Peak shifts ~1eV to higher binding energy.



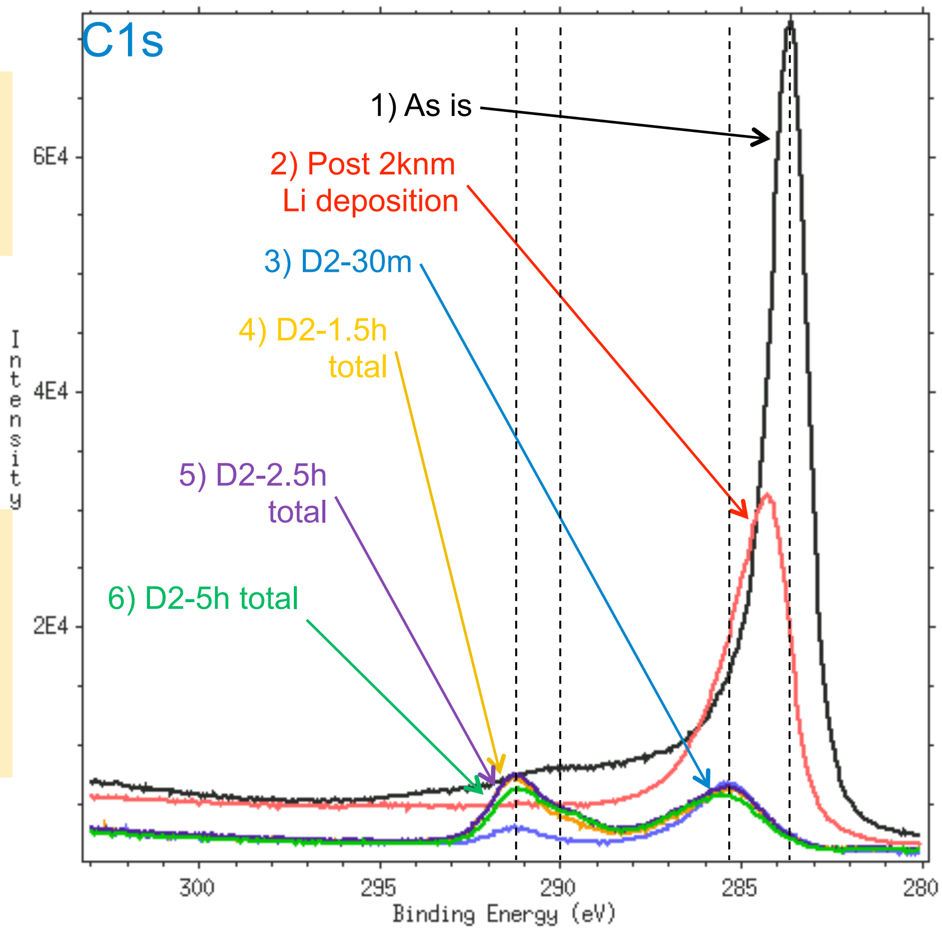

3) 30 minute deuterium irradiation ($\Gamma \approx 1.5$ E15 $cm^{-2}$) causes a new peak to develop at 291 eV. The 284 eV peak shifts again to higher binding energy, now residing ~285 eV.

4) The relative intensity of the 291 eV peak compared to the 529.5 eV peak increases with subsequent irradiations. Peak at 285 eV ceases to change.

# Results – Li-D-O and C functionality

1) ATJ graphite shows a graphitic C1s peak at 284 eV. Carbonate presence is observed at 290 eV.

2) Lithium deposition results causes the FWHM of the primary peak to increase. Peak shifts ~1eV to higher binding energy.




3) 30 minute deuterium irradiation ($\Gamma \approx 1.5$ E15 $cm^{-2}$) causes a new peak to develop at 291 eV. The 284 eV peak shifts again to higher binding energy, now residing ~285 eV.

4) The relative intensity of the 291 eV peak compared to the 529.5 eV peak increases with subsequent irradiations. Peak at 285 eV ceases to change.

5,6) Change of relative intensity slows at some D fluence threshold.

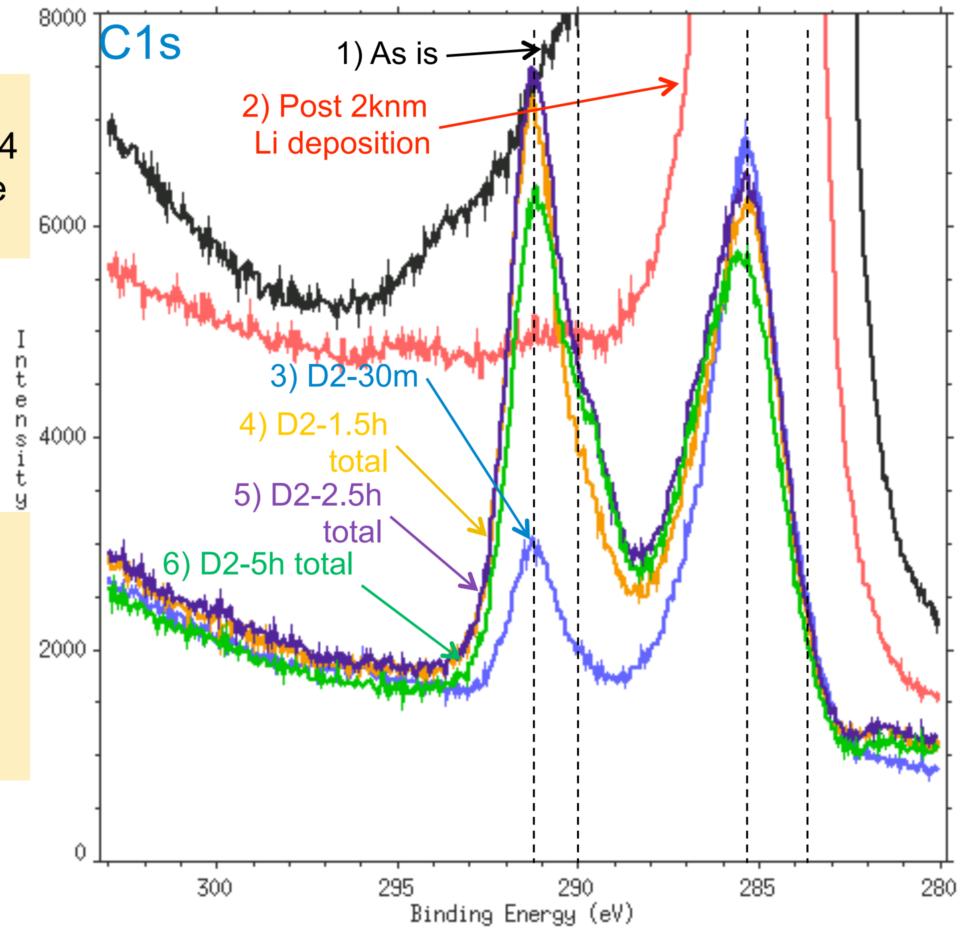


PURDUE ENGINEERING

# Results – Li-D-O and C functionality

1) ATJ graphite shows a graphitic C1s peak at 284 eV. Carbonate presence is observed at 290 eV.

2) Lithium deposition results causes the FWHM of the primary peak to increase. Peak shifts ~1eV to higher binding energy.




3) 30 minute deuterium irradiation (Γ ≈ 1.5 E15 $cm^{-2}$) causes a new peak to develop at 291 eV. The 284 eV peak shifts again to higher binding energy, now residing ~285 eV.

4) The relative intensity of the 291 eV peak compared to the 529.5 eV peak increases with subsequent irradiations. Peak at 285 eV ceases to change.

5,6) Change of relative intensity slows at some D fluence threshold.

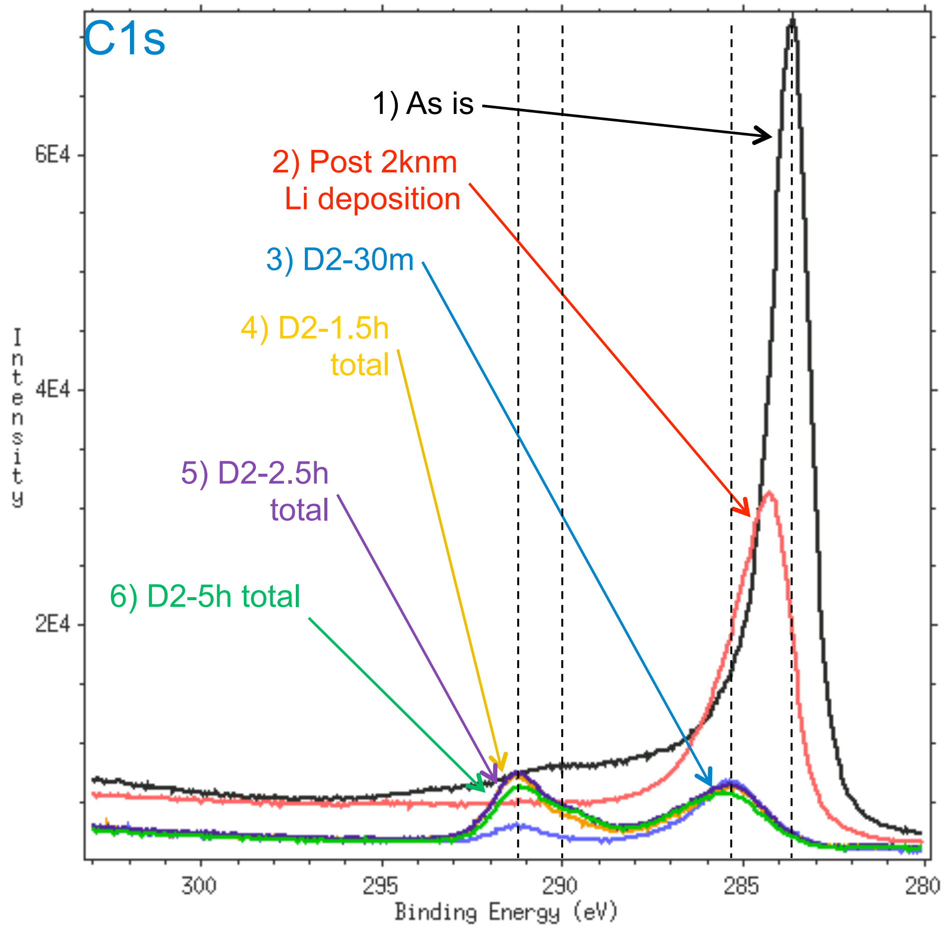


PURDUE ENGINEERING

# Results – Li-D-O and C functionality

**Observations**

Based on these results and control experiments



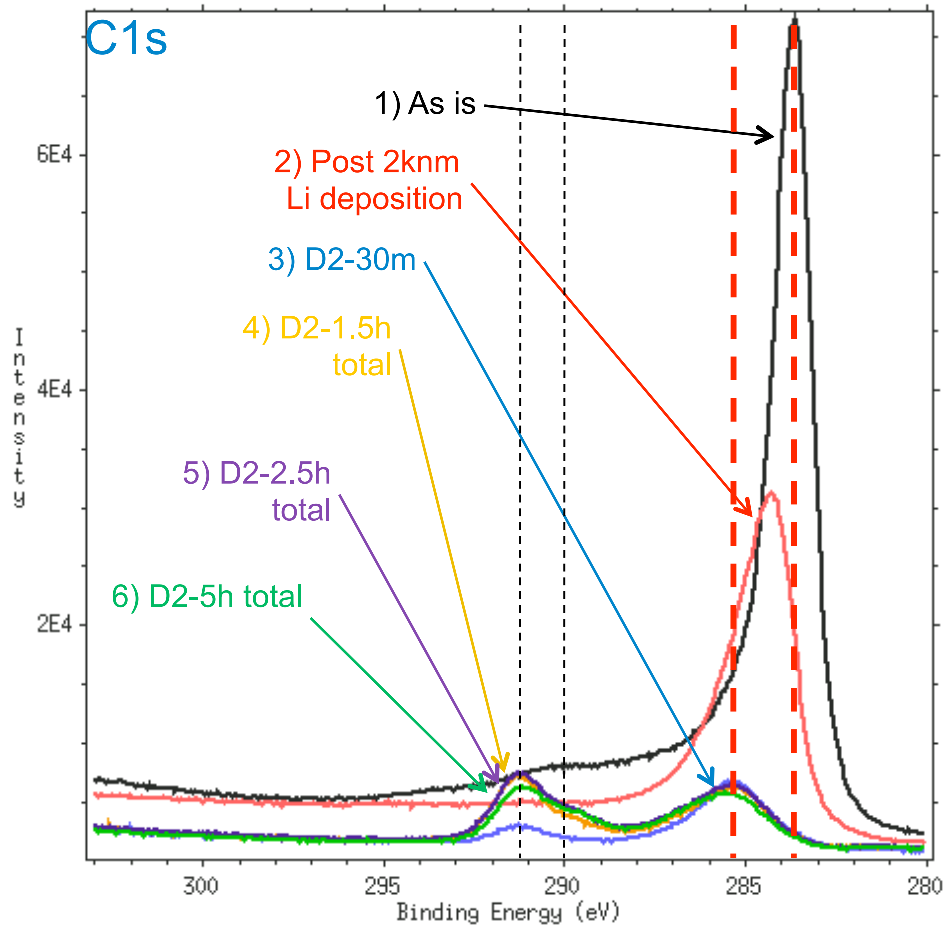



PURDUE ENGINEERING

# Results – Li-D-O and C functionality

## Observations

Based on these results and control experiments




**284-285 eV**

- Control experiments have shown that 2 peaks momentarily coexist.
- Development of new peak indicates new bonding functionality.

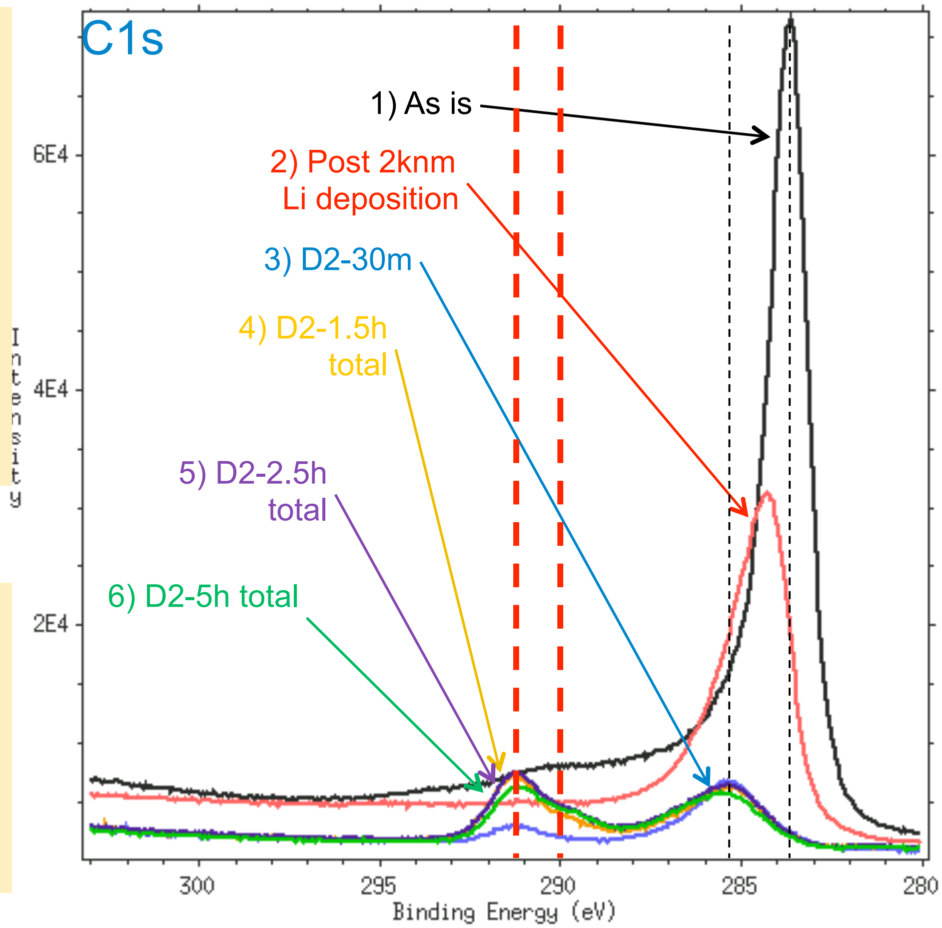
# Results – Li-D-O and C functionality

## Observations

Based on these results and control experiments

**291 eV**
- Only develops after irradiating a *lithiated* sample.
- Relative intensity increases with higher D2 fluence.
- Eventually peak "saturates" and does not respond to increased D fluence.

**290 eV**
- Slight carbonate influence observed.
- Air exposure of a lithiated sample results in a carbonate peak (not shown).




**284-285 eV**
- Control experiments have shown that 2 peaks momentarily coexist.
- Development of new peak indicates new bonding functionality.

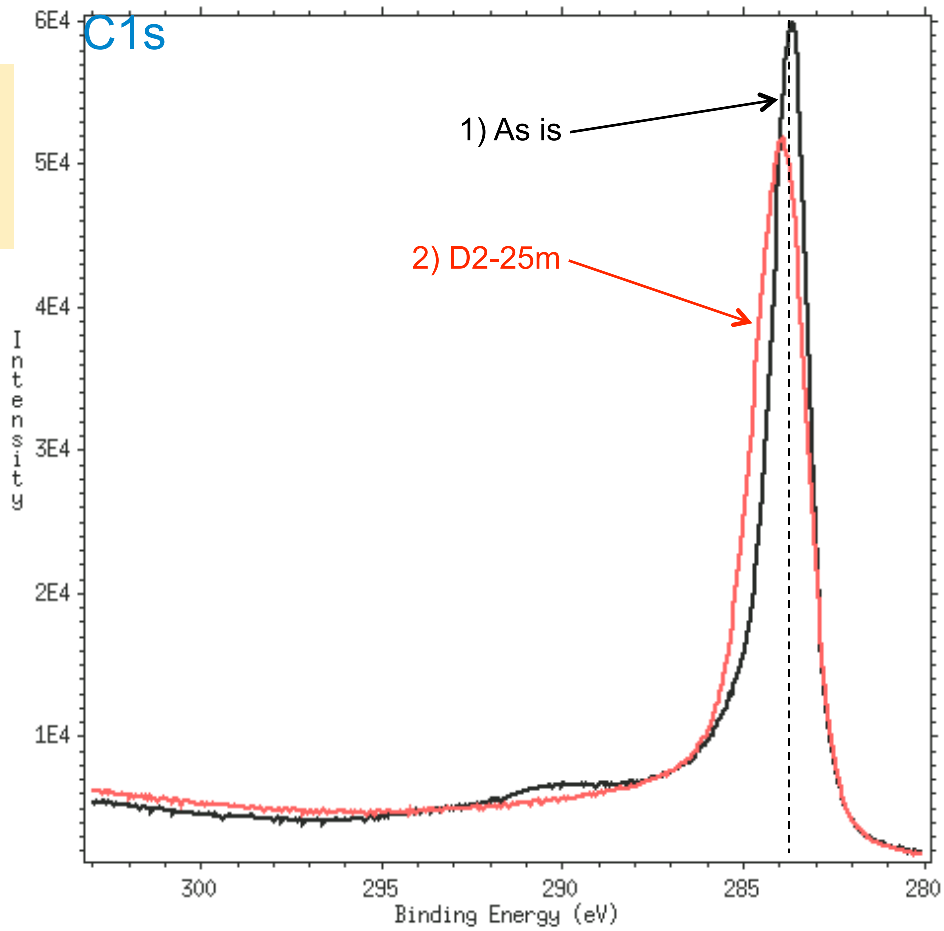


PURDUE ENGINEERING

# Results – Li-D-O and C functionality

## Control experiments

**Procedure (repeat):**
ATJ graphite was irradiated with D *without* any lithium conditioning.




**Result:**
Graphitic peak (284 eV) shifted slightly to higher binding energy.

Carbonate peak (290 eV) diminished.

No new peaks were observed.

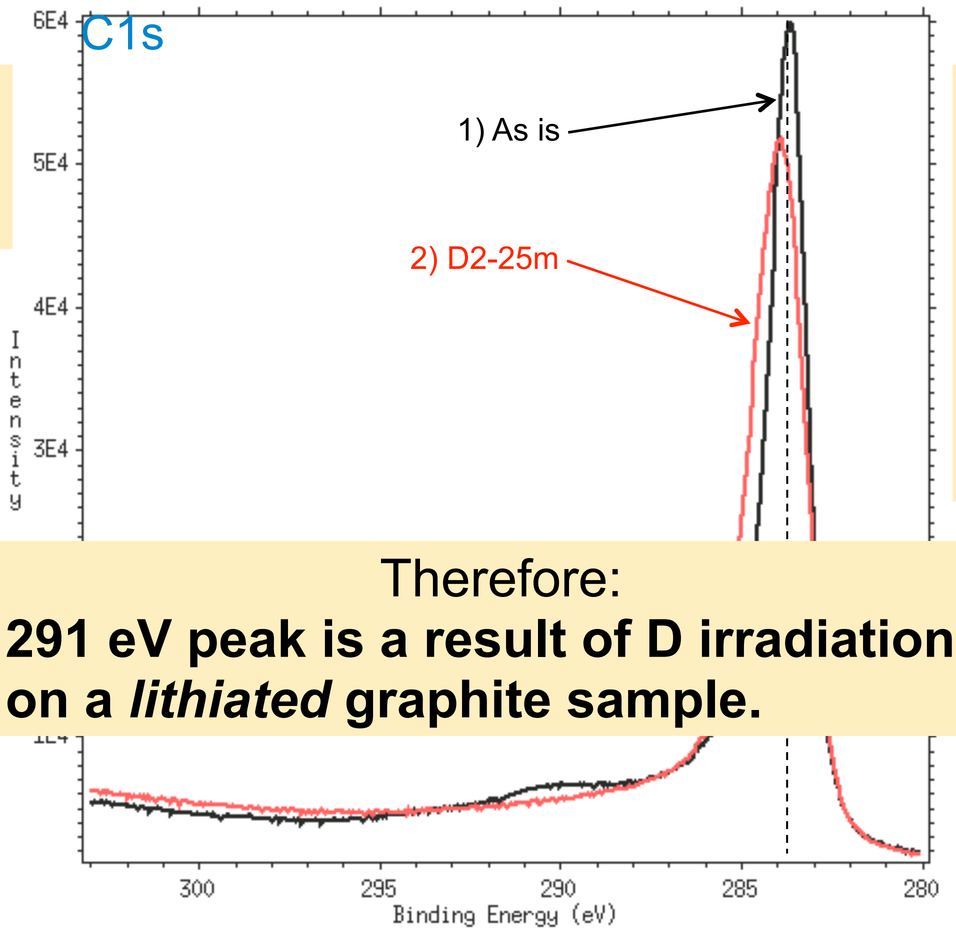


PURDUE ENGINEERING

# Results – Li-D-O and C functionality

## Control experiments

**Procedure (repeat):**
ATJ graphite was irradiated with D *without* any lithium conditioning.




**Result:**
Graphitic peak (284 eV) shifted slightly to higher binding energy.

Carbonate peak (290 eV) diminished.

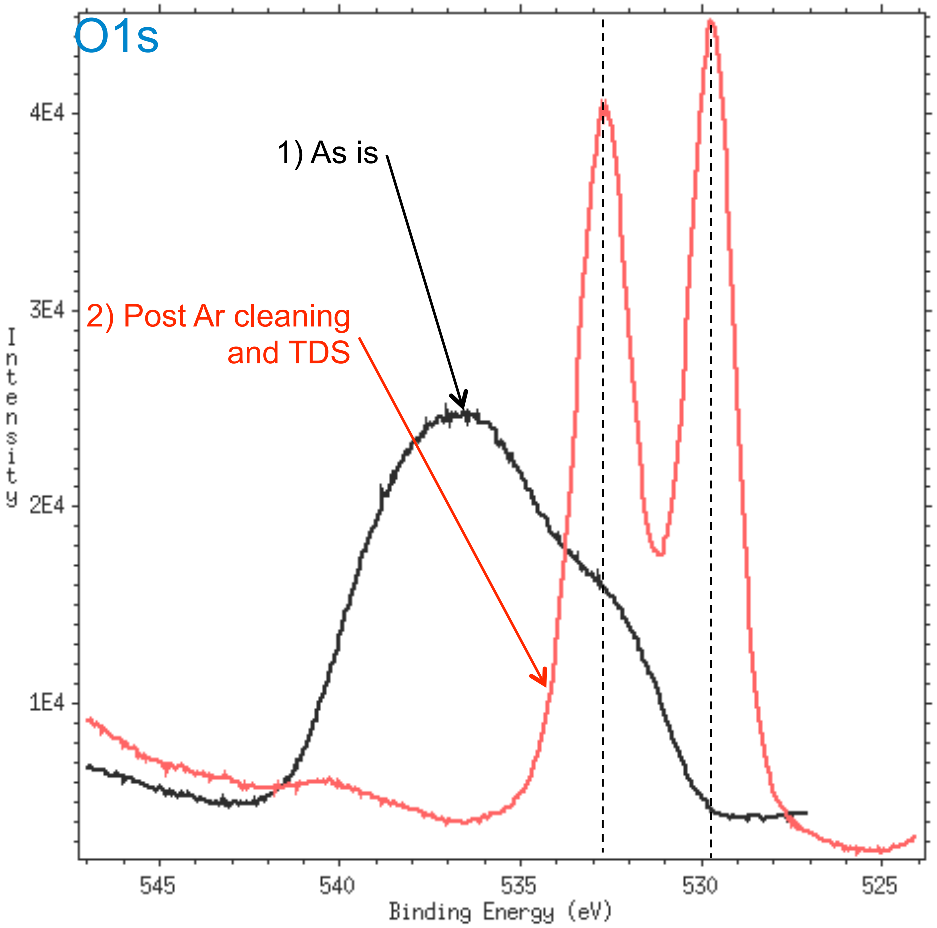
No new peaks were observed.

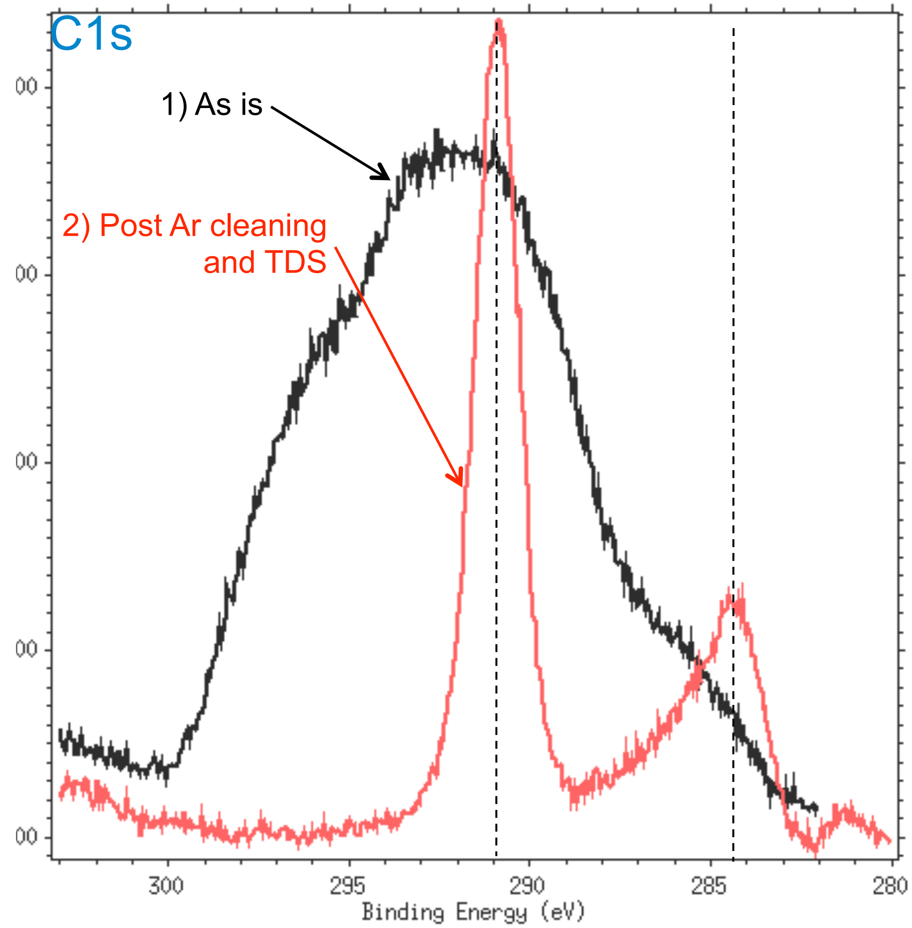
Therefore:
**291 eV peak is a result of D irradiation on a *lithiated* graphite sample.**



PURDUE ENGINEERING

# Results – Post mortem NSTX FY08 tiles




Treatment procedure results in peaks at 529.5 and 533 eV.

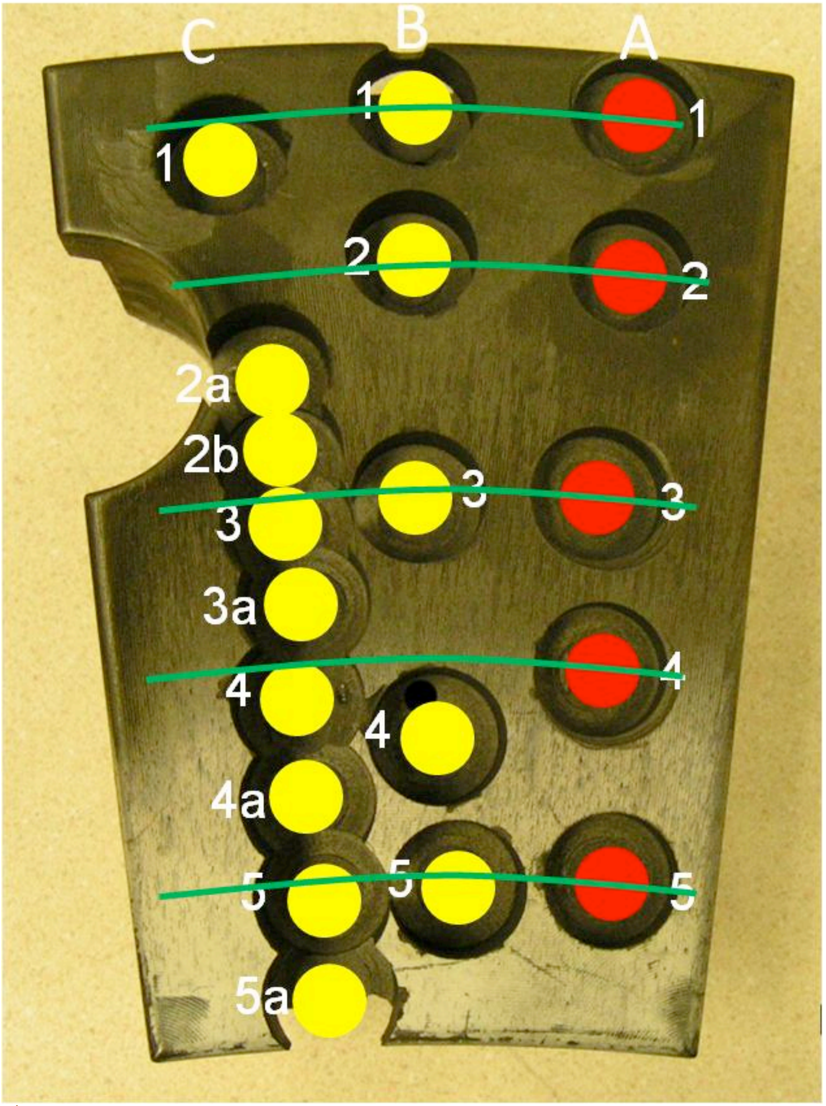
Treatment procedure results in peaks at 284 and 291 eV.

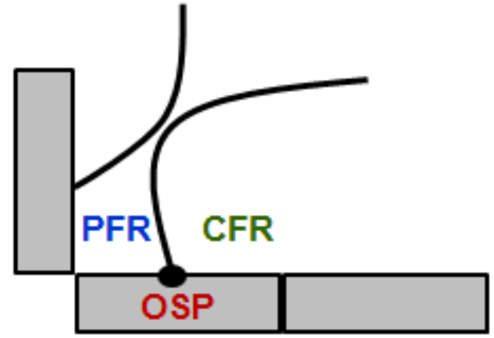
Before treatment procedure, passivated tiles exhibit **broad** peaks.
After cleaning, tiles resemble peaks found in control experiments.



PURDUE ENGINEERING

# *NSTX Tile A408-002*




Common Flux Region (CFR)

Outer Strike Point (OSP)

Private Flux Region (PFR)

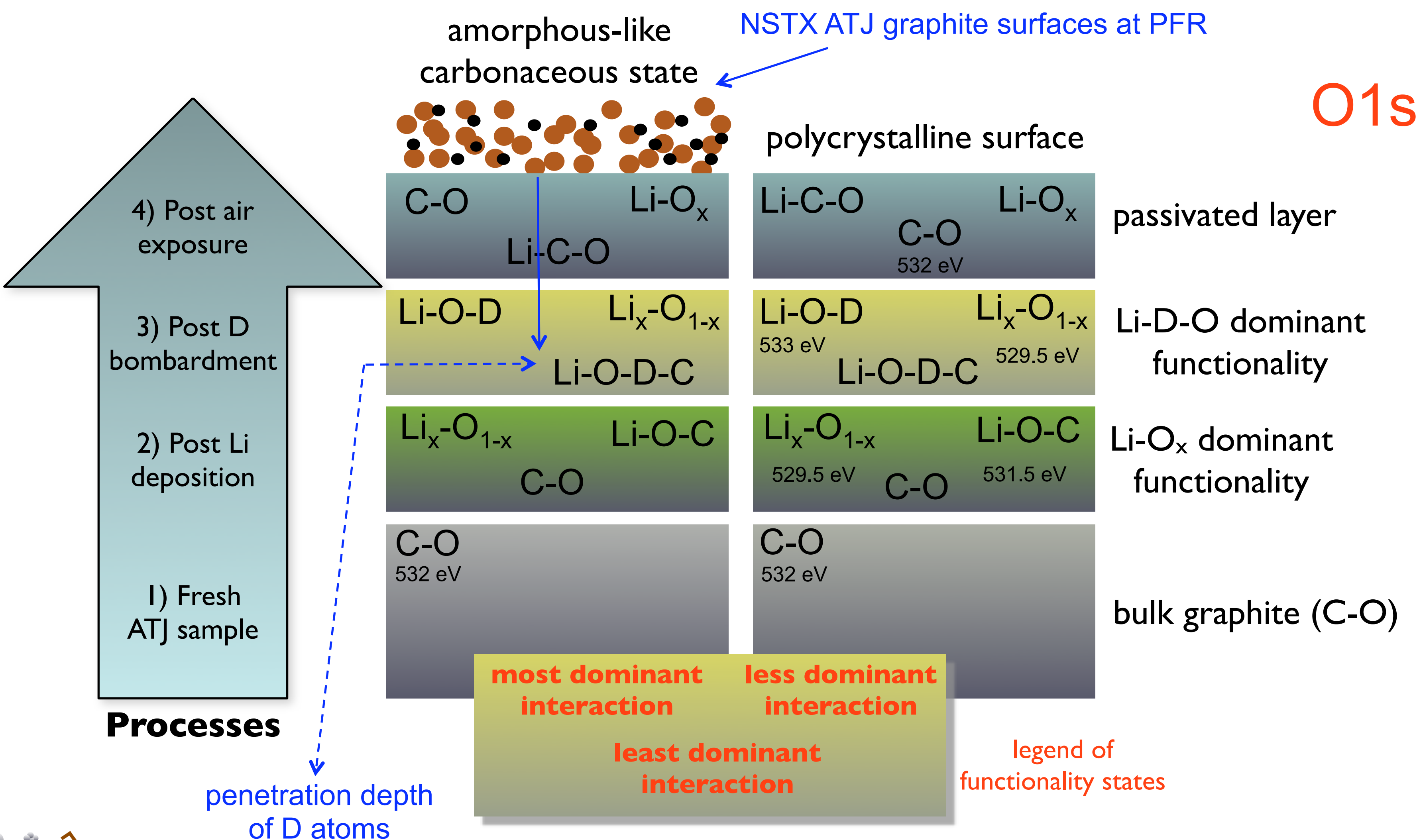
# *Summary of controlled in-situ XPS studies*

- Oxygen
  - Li and O interactions, on a graphite substrate, are manifest at 529.5 eV in the XPS spectrum. Peak diminishes with larger D fluence.
  - Li, O, and D interactions, on a graphite substrate, are manifest at 533 eV. Peak dominates with larger D fluence.
- Carbon
  - Li, D, and C interactions are manifest at 291 eV. Relative peak energy increases with increased D fluence. Changes cease to occur at a yet to be discovered D fluence threshold.
- Post-mortem tiles
  - Treatment (Ar sputtering and heating) changes passivated, broad, inconsistent peaks to align with consistently produced peaks found in controlled experiments.
  - "Broad" peaks consistent with a highly porous and amorphous carbonaceous layer (in time-integrated PFR region)



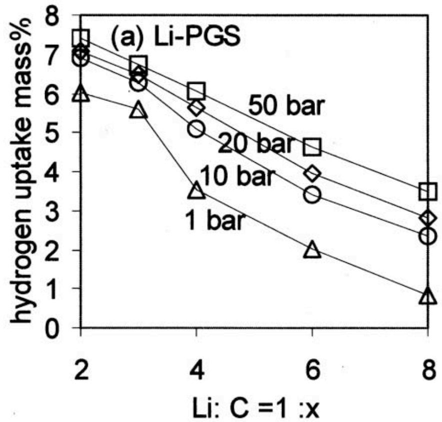
PURDUE ENGINEERING

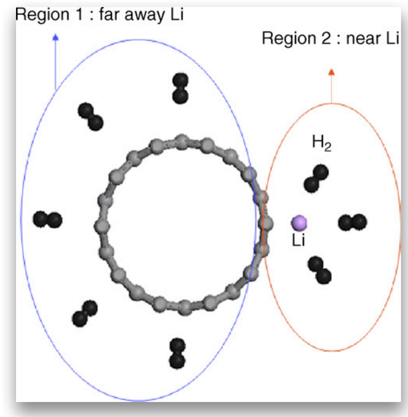
# *"Current" qualitative hypothesis of functionality states of lithiated-graphite surfaces in NSTX*



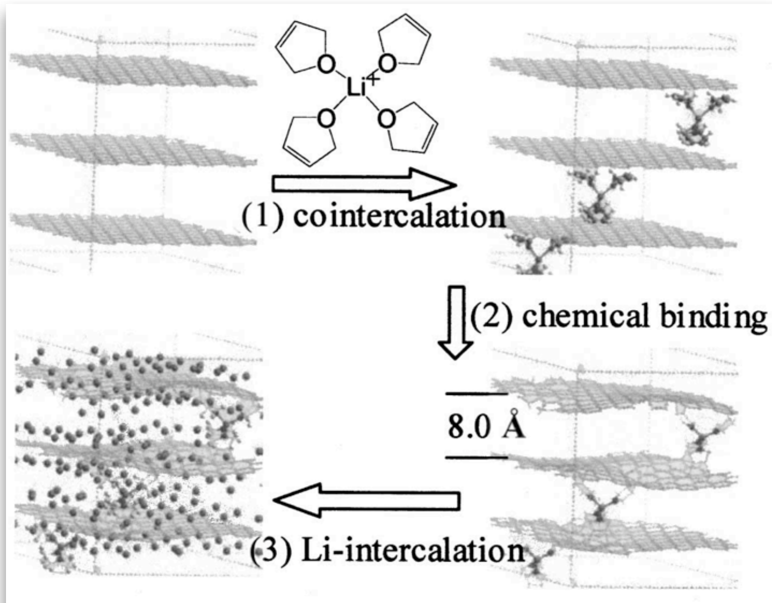
# Mechanisms for D retention in lithiated ATJ graphite surfaces

- Structural diversity in carbon leads to a number of "functionalities" or "preferred interactions" between hydrogen and Li in a carbon matrix
- Literature in the Li-C-H system is consistent with our observations
- Disorder in the carbon matrix can leave a large number of C valences unsaturated as dangling bonds
- Li can also bind in the *vicinity* of H atoms
- Electronic transfer from Li to C atoms can induce dipole interactions with H
- More Li, more H interaction and effectively higher retention




Lithium doping in nano-structured carbon surfaces using DFT and QMD modeling[2,3]



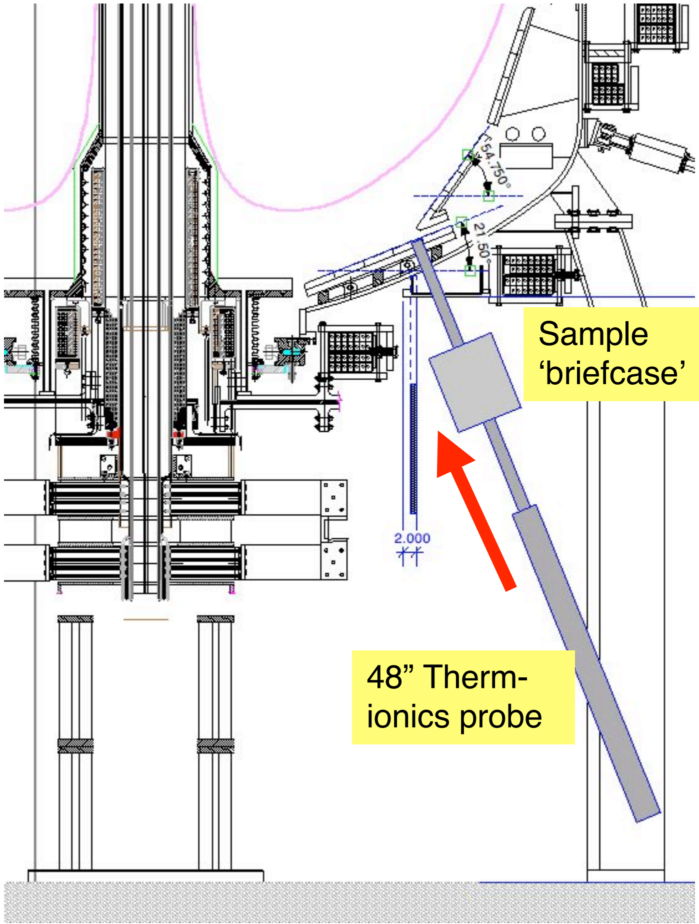


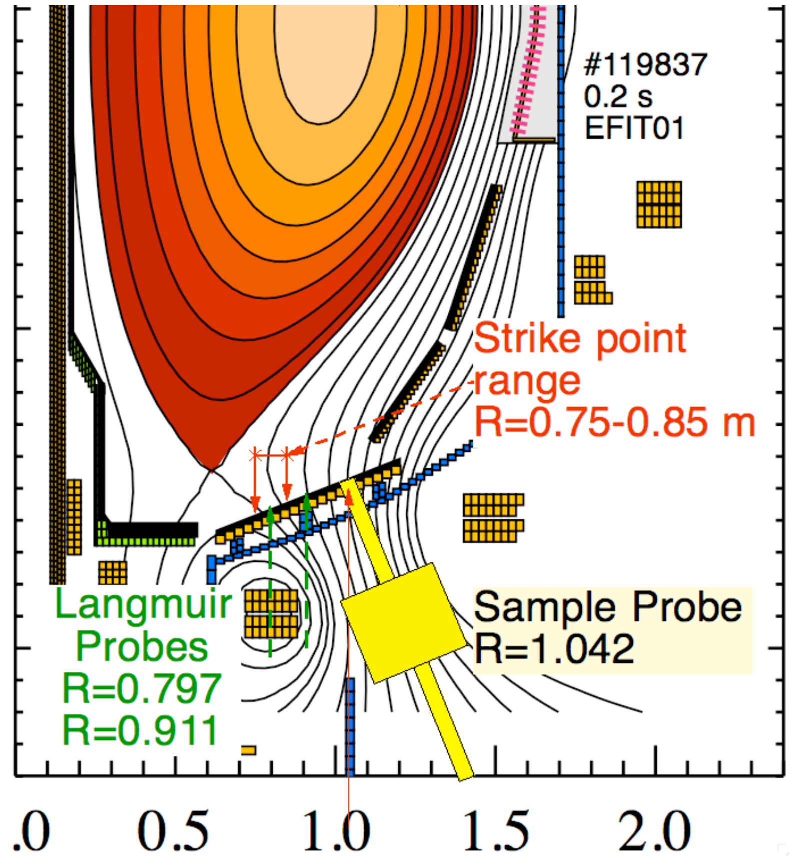
[1]J.R. Dahn et al. Science 270, October 1995, 590
[2]W.Q. Deng et al. Phys. Rev. Lett. 92, 2004, 166103
[3]J.H. Cho et al. Catalysis Today, 120, 2007, 407




PURDUE ENGINEERING

# NSTX PMI Probe




Sample Probe aims to address: *"fundamental processes governing particle balance...using lithium surfaces in the divertor…"* (Joule milestone language)

FY'09 Thermal Desorption Spectroscopy ex-vessel, promptly after plasma exposure (no air exposure).

# *Summary of PMI Probe experiments*

## ***With no lithium conditioning***

**Neutral Beam Plasmas**

- ATJ132 – TDS at NSTX
- ATJ133 – TDS at Purdue
- Pd425 – XPS
- Si105

**Ohmic Heated Plasmas**

- ATJ134 – TDS at NSTX
- ATJ135 – TDS at Purdue
- Rh sample
- Si112

## ***With lithium conditioning***

**Neutral Beam Plasmas**

- ATJ138 – TDS at NSTX
- ATJ139 – TDS at Purdue
- Pd431 – XPS
- Si109

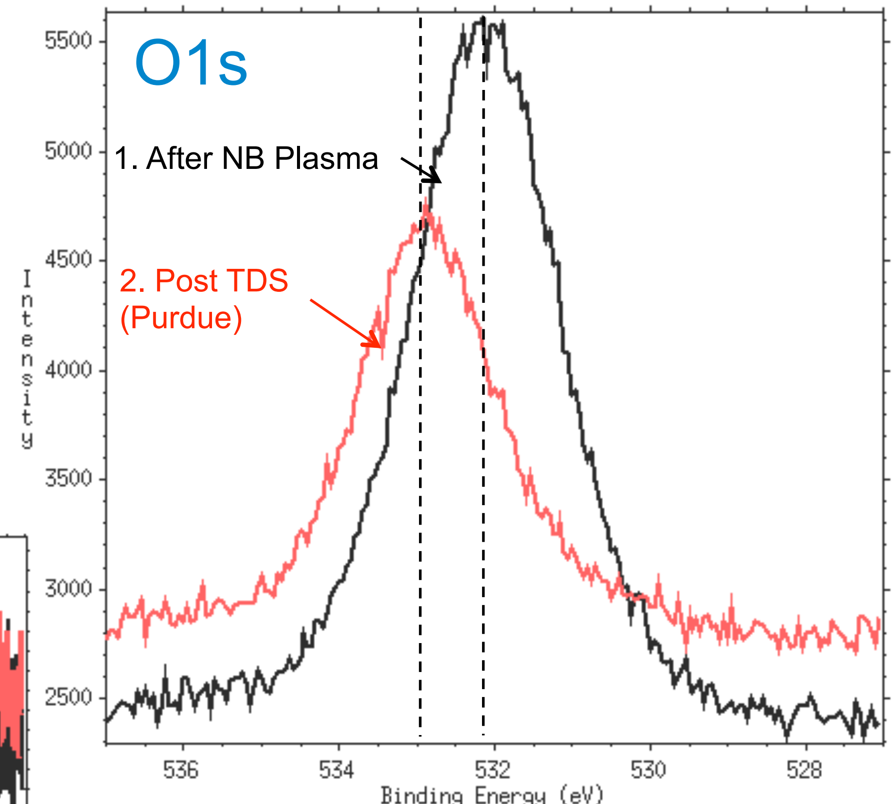
**Ohmic Heated Plasmas**

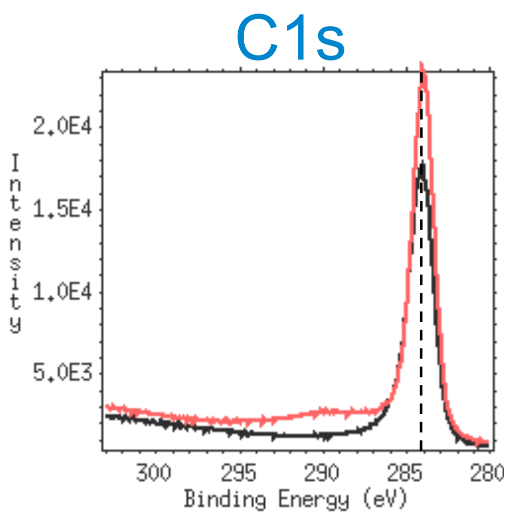
- ATJ136 – TDS at Purdue
- ATJ137 – TDS at Purdue
- Pd422 – XPS
- Si108

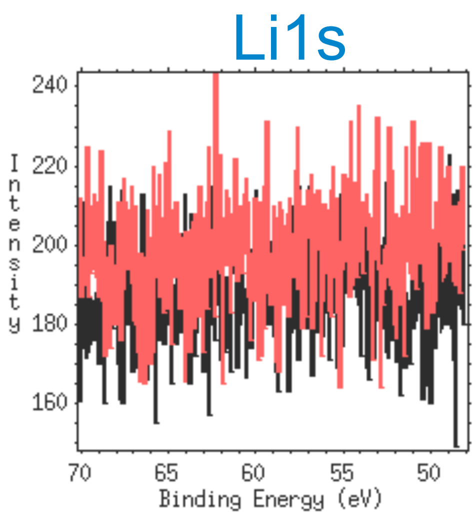
# ATJ133 – Exposed to NB Plasma

Brief DoE

- No lithium conditioning
- 6 NSTX NB plasma shots
- Shipped to Purdue under Ar
- TDS at Purdue



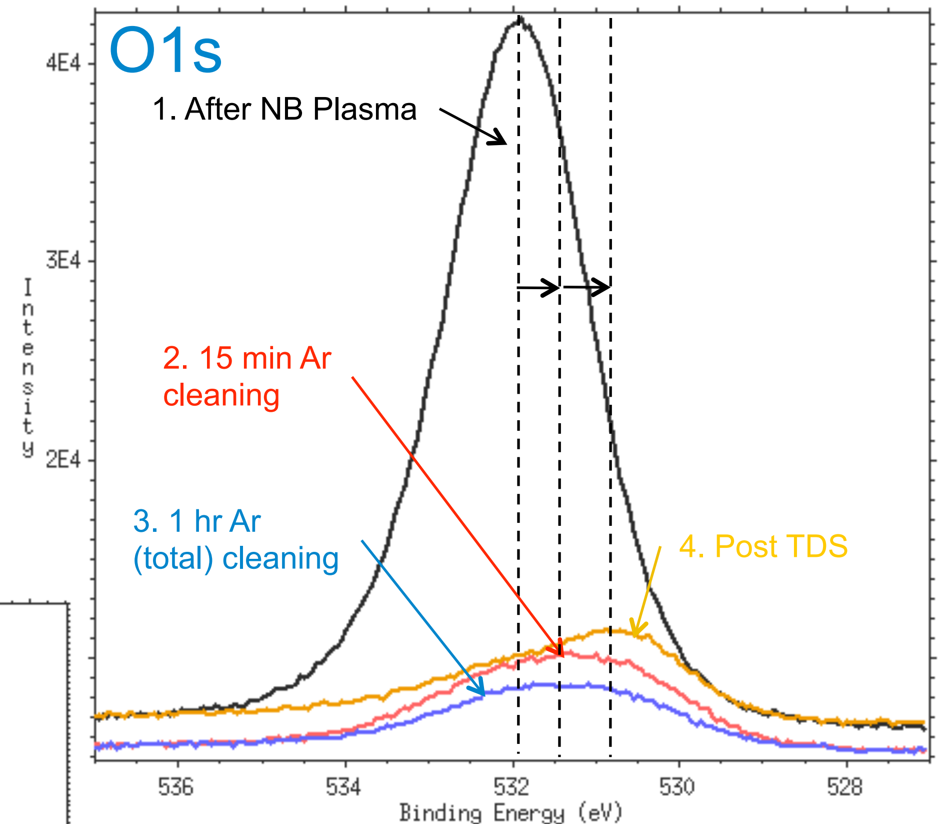



# ATJ139 – Exposed to NB Plasma

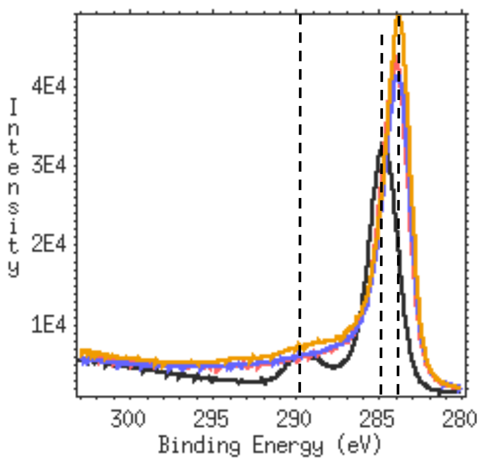
Brief DoE

- Lithium conditioning
- 6 NSTX NB plasma shots
- Ar cleaning
- TDS performed at Purdue
- XPS at Purdue

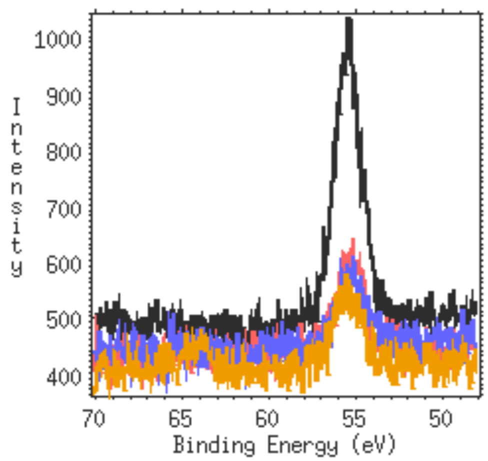



Note: 30 min Ar cleaning XPS scan was also taken

C1s




Li1s




RSSEL

PURDUE ENGINEERING

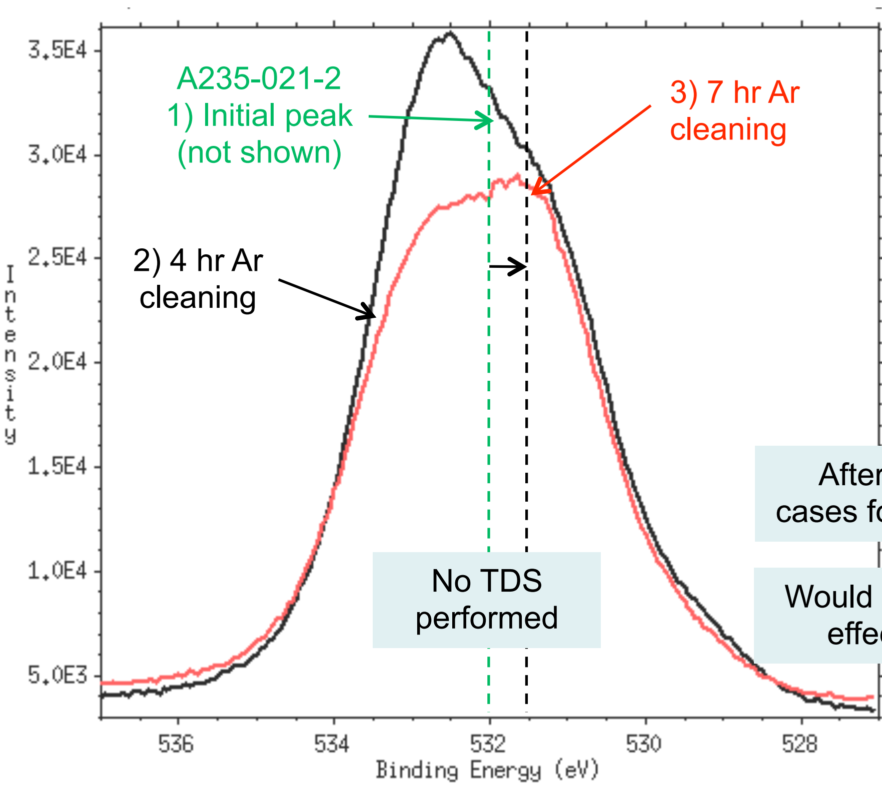
# AJT139 vs. post-mortem tile near LITER

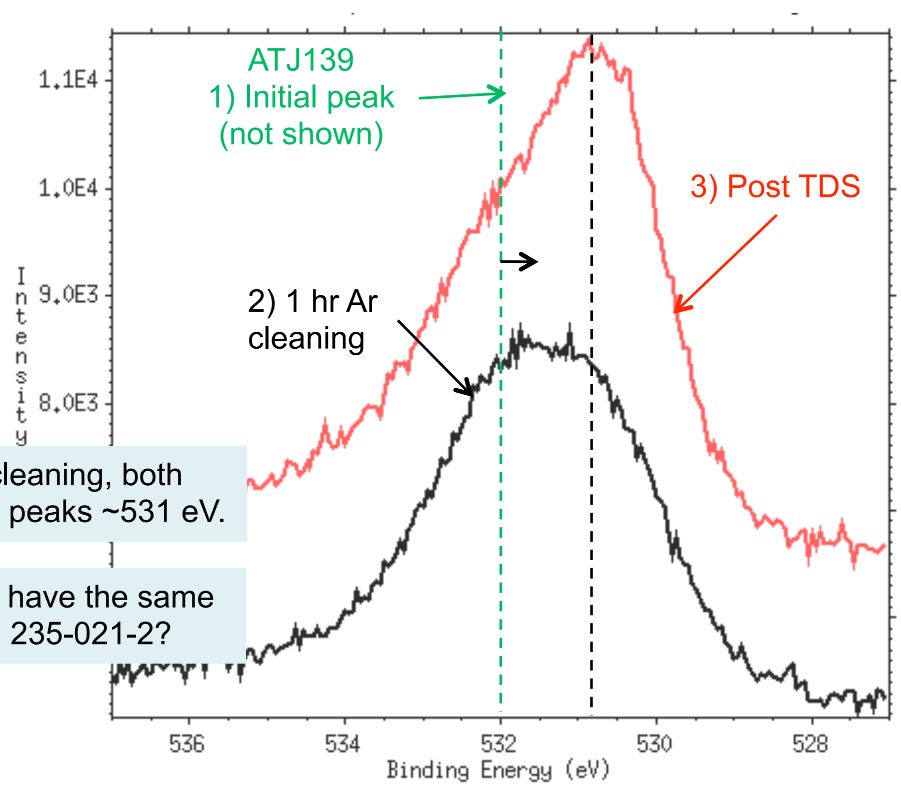
## NSTX Tile A235-021-2

- Staged Ar cleaning

## ATJ139

- Lithium conditioning
- 6 NSTX NB plasma shots
- Ar cleaning
- TDS performed at Purdue




After Ar cleaning, both cases found peaks ~531 eV.

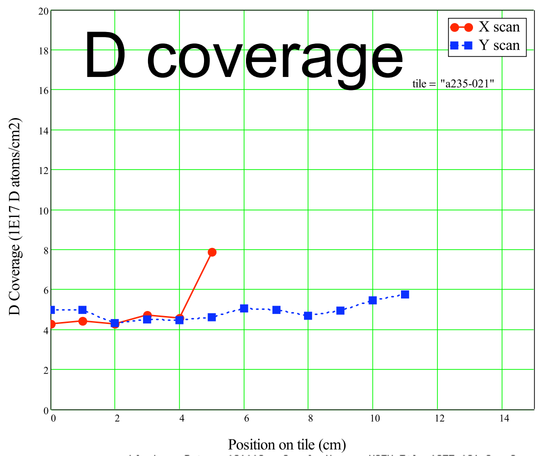
Would TDS have the same effect on 235-021-2?

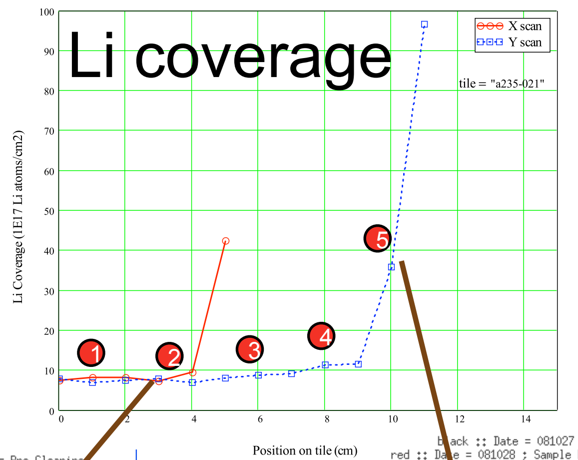
RSSEL

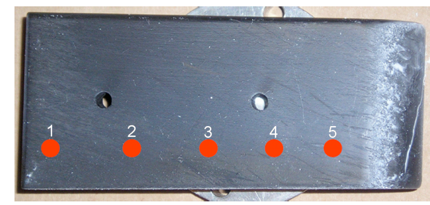
PURDUE ENGINEERING

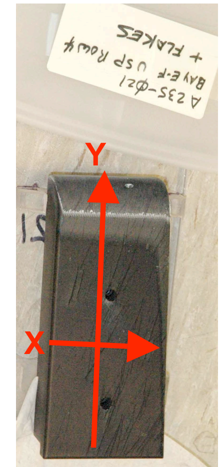
# Comparisons of Ion Beam data with XPS

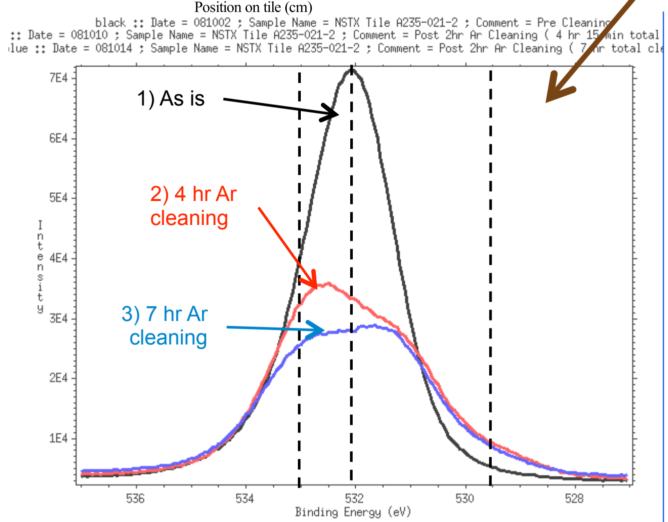
- Lithium dependence on surface chemistry

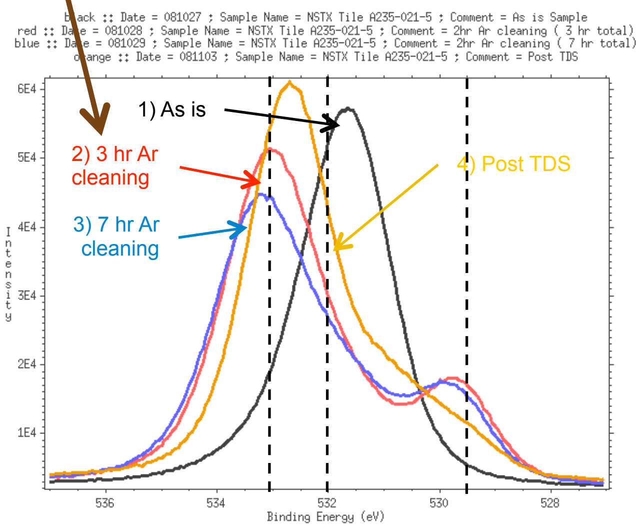


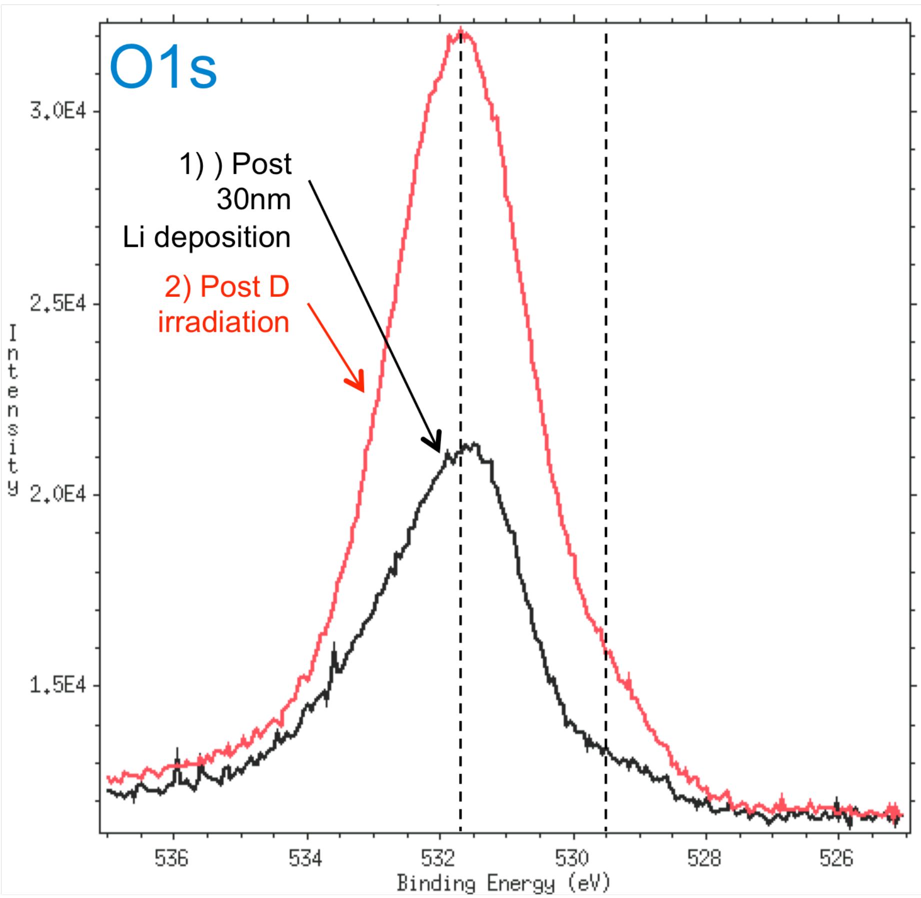



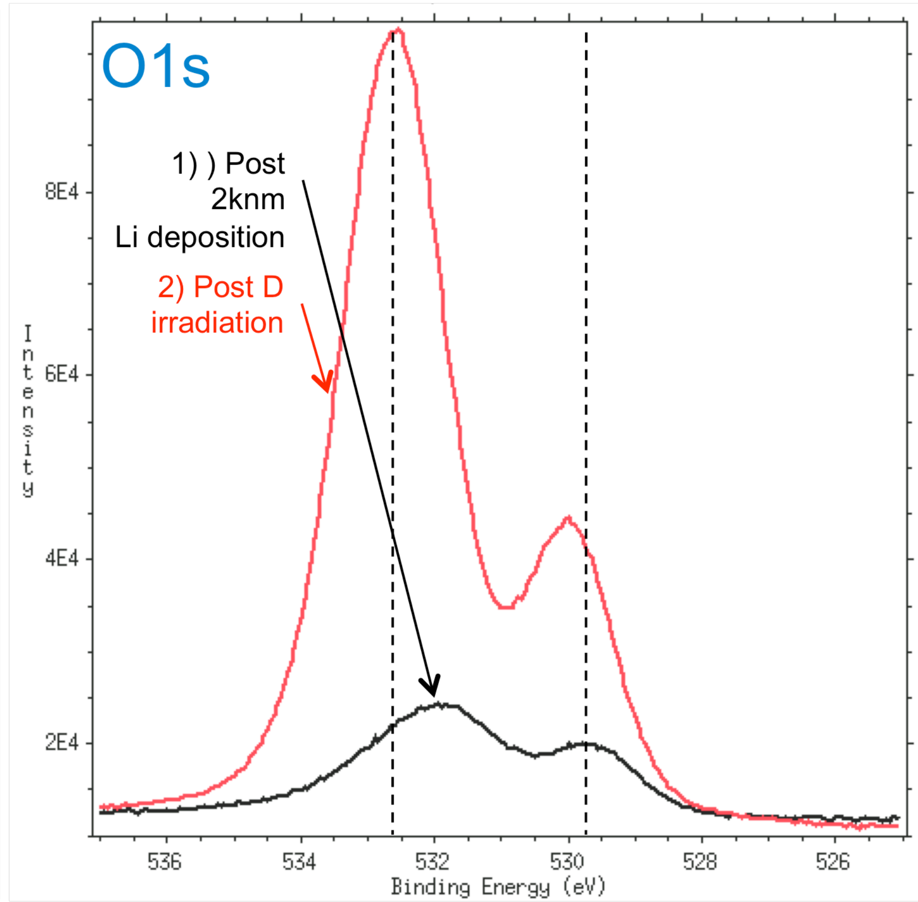




Ion Beam Analysis of Li and D on Tiles from NSTX, W. Wampler, 2006

NSTX Tile A235-021

# *Lithium dose affects Li-D-O-C functionality*



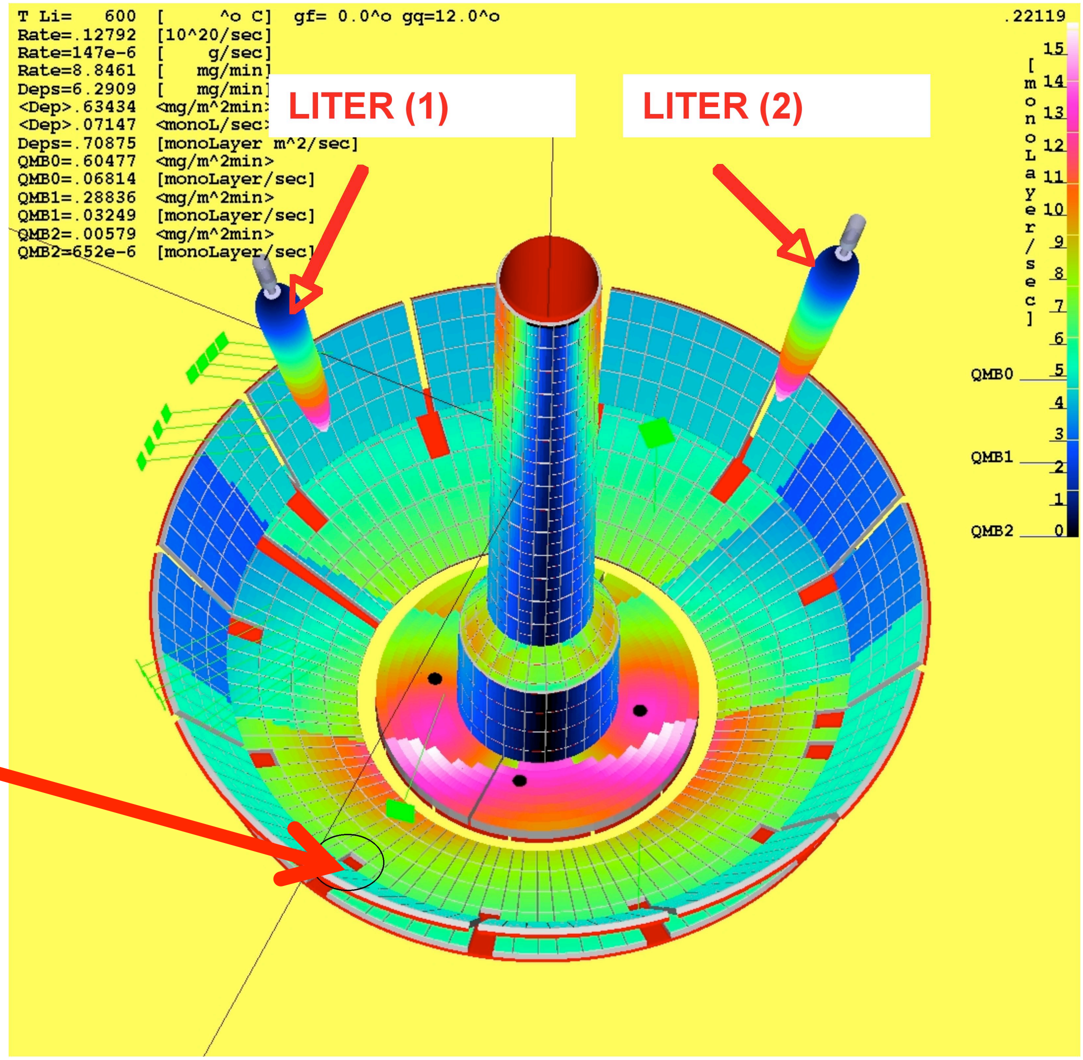

Li-30nm post deposition, post D irradiation

Li-2000 nm post deposition, post D irradiation

RSSEL

PURDUE ENGINEERING

# *NSTX PMI Probe location and lithium deposition*




PMI Probe location

Simulation by Leonid Zakharov

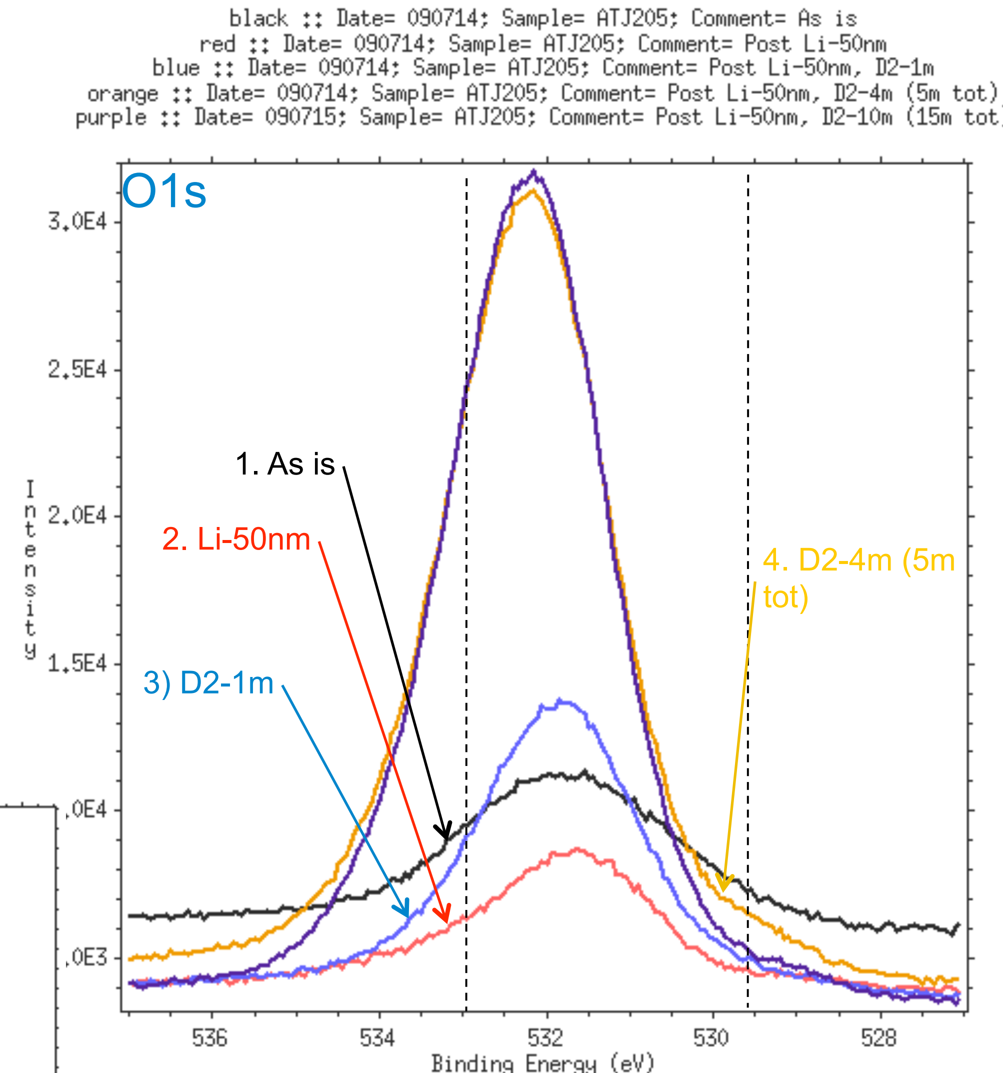
RSSEL

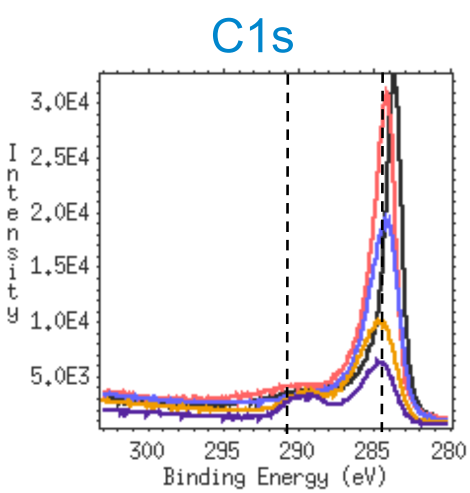
PURDUE ENGINEERING

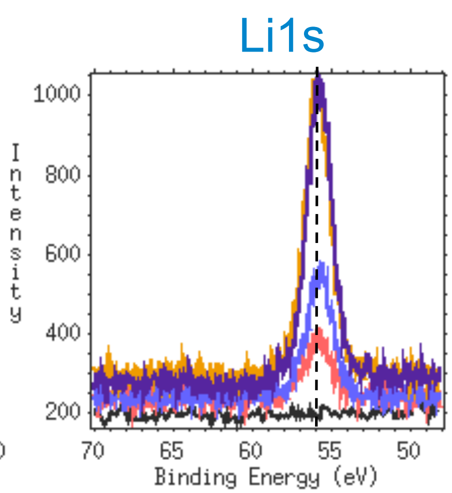
# *ATJ205*

Brief DoE

- Li-50nm deposition
- D2-1 minute
- D2-4 minutes to 5m total



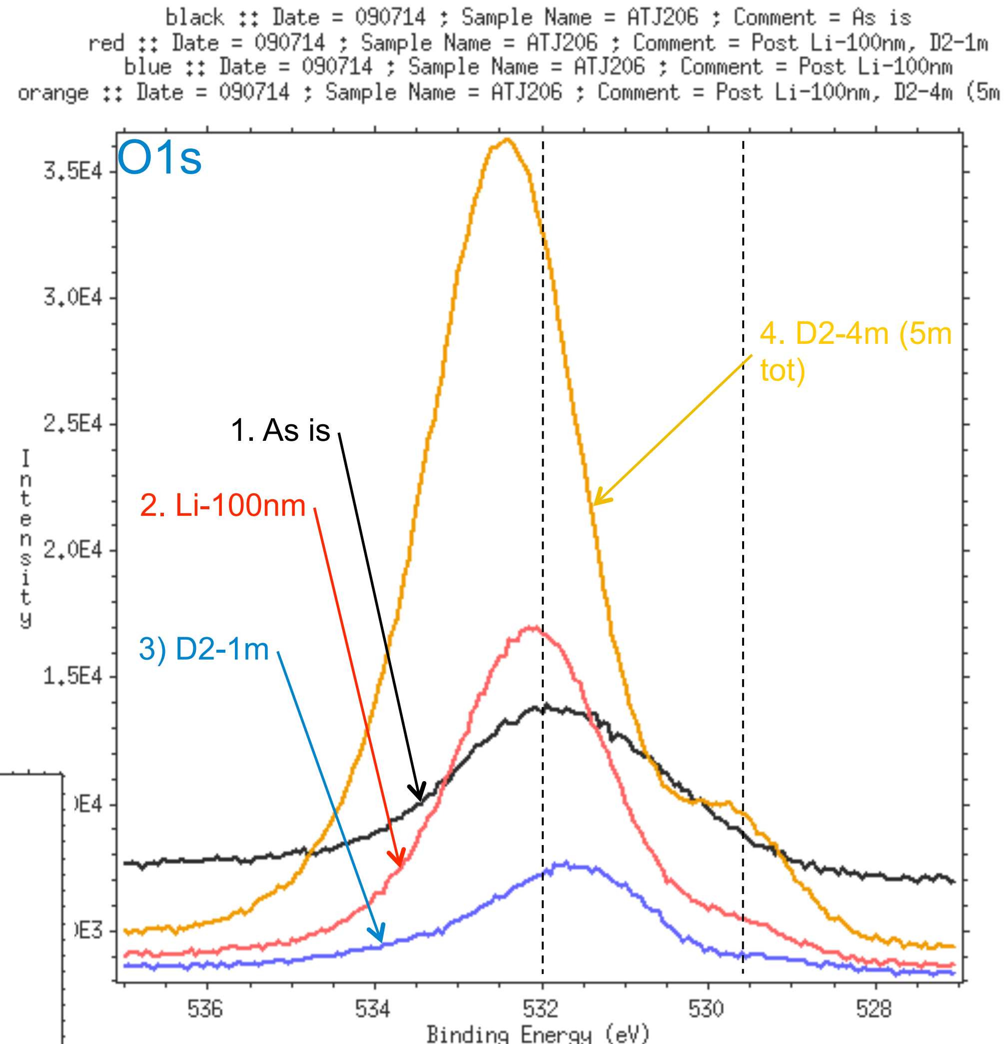



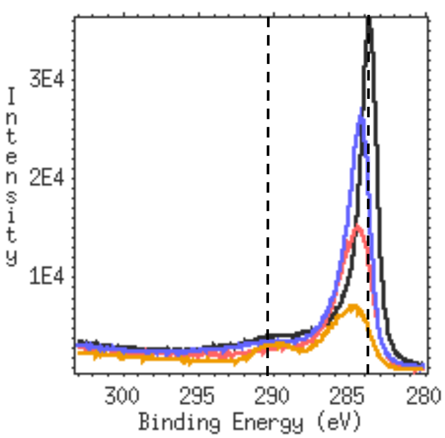
# *ATJ206*

Brief DoE

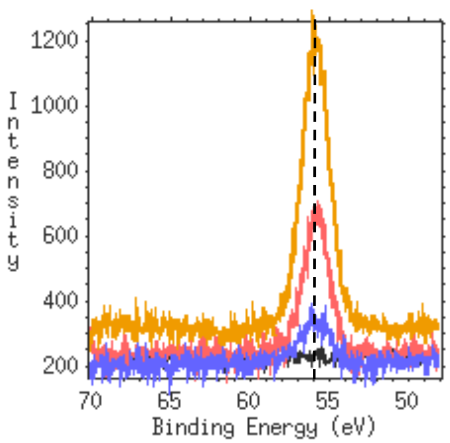
- Li-100nm deposition
- D2-1 minute
- D2-4 minutes to 5m total




C1s




Li1s




Li deposition does not produce substantial peak at 529.5 eV (slight shoulder). D2-1m causes immediate shift, though not fully to 533 eV. Larger fluence accentuates the presence of 529.5 eV

PURDUE ENGINEERING

# PMI Probe sample examination

- April 22
  - Shots 132973-133018
  - XP911 occupied 8 Ohmic plasma shots
  - Assume Li coverage: 25% of 40m$^2$ area in vessel
  - In 8 shots, 446 mg deposited (84 nm)
- SEM of Si sample shows < 500-nm film
- Pd425
  - No Li conditioning
  - Exposed to 6 NB plasmas
  - Post analysis 4-point probe showed a D concentration of ~5.16 x 10$^{20}$ m$^{-2}$
  - Pd sample was heated beyond 200 C emitting implanted D
  - Langmuir probes showed average deuterium flux of: ~3.34 x 10$^{22}$ m$^{-2}$

Si108

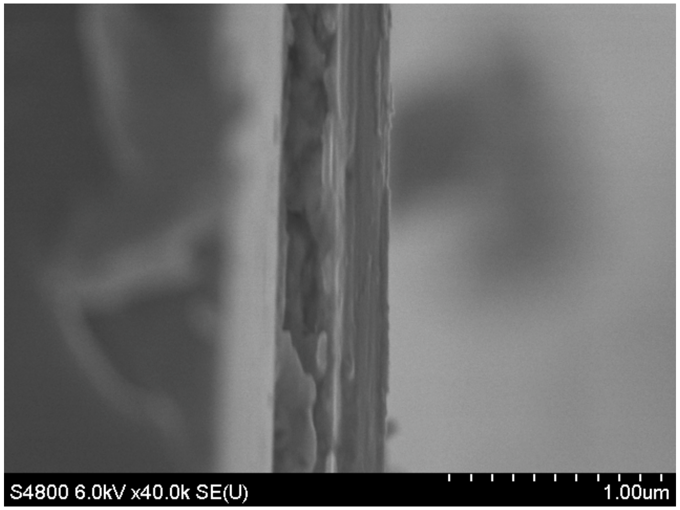



Fig. 1 Sample probe with ATJ graphite, Si and Pd samples

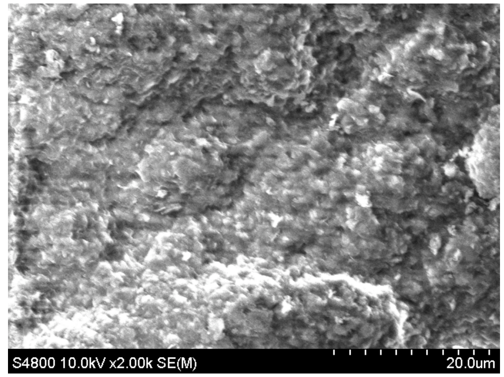
PURDUE ENGINEERING

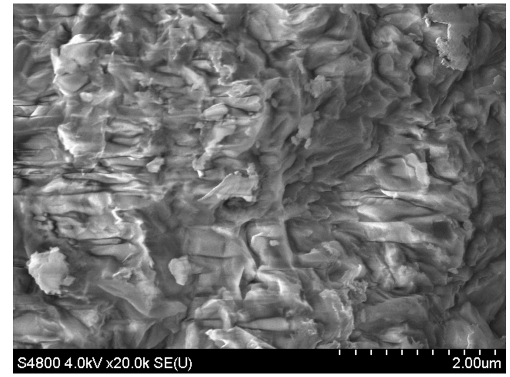
# *Surface morphology of ATJ graphite surfaces*

Low magnification | High magnification

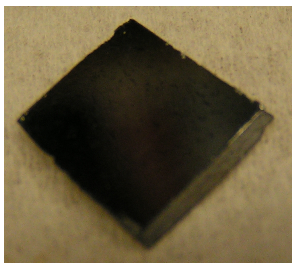
## NSTX post mortem tile

**Tile A408-002-C5**
Removed after FY08 campaign

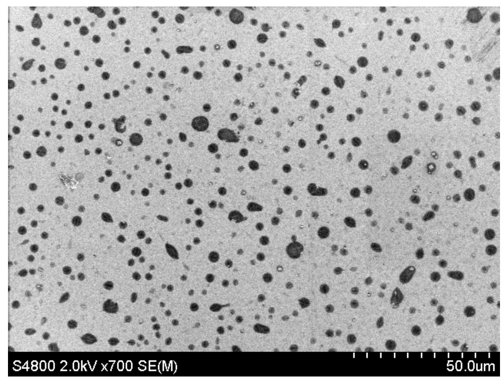


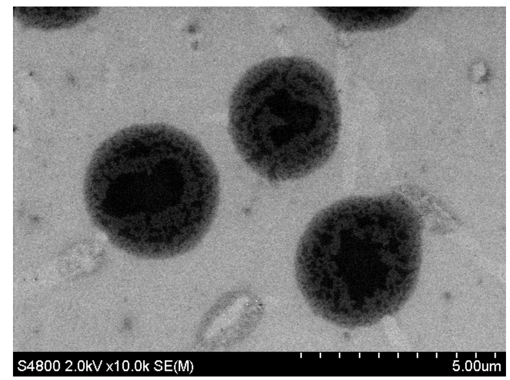




## Si probe sample



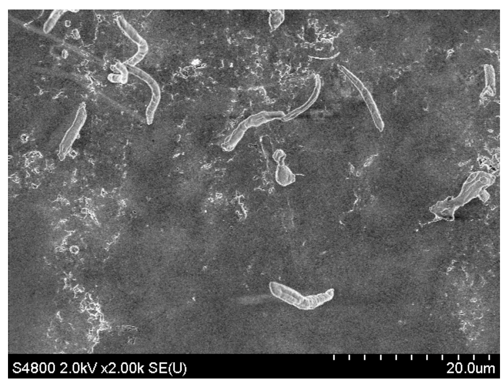
**Si108**
Exposed to 8 NSTX Ohmic plasmas via sample probe

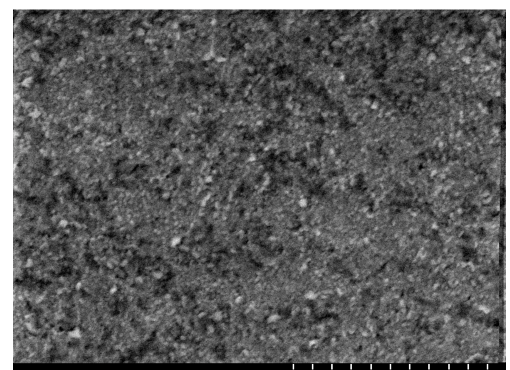



## Control graphite sample

**ATJ147a**
2000 nm Li deposited, 1.5 hr D irradiation





PURDUE ENGINEERING

# *Implications for LITER and LLD operation*

- Controlled *in-situ* surface analysis of lithiated ATJ graphite surfaces show:
  - initially Li readily intercalates
  - Over time with large lithium dose (and with D) a diffusion barrier is created slowing intercalation to bulk
  - D irradiation and oxidation can also drive Li to surface
- It is obvious that "*the more lithium the better*"
  - Our work shows mechanism for D retention dependent on charge transfer mechanisms in Li:C:D and also on carbon structure (morphology)
  - Spreading more lithium on carbonaceous surfaces with thicknesses of at least 400-500 nm show signs of D retention (LLD will help with this)

Acknowledgements

D. Zemlyanov, Birck Nanotechnology Center

S.S. Harilal, Purdue University

M. Nieto, M.R. Hendricks, V. Titov, P. Plotkin

J.N. Brooks, Purdue University

C. Skinner, H. Kugel, R. Majeski, R. Kaita
*Princeton Plasma Physics Laboratory*

- Purdue work under US-DOE Contract DE-FG02-08ER54990